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NASA CR 51858

N63 22882

CODE-1

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XEROX $ 6.60 ph

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Pratt & Whitney Aircraft

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EAST HARTFORD 8, CONNECTICUT

TITLE CAPS

Progress Report, Determination of the Emissivity of Materials

Report No. PWA-2255

(See cover

Report Period: January 1 through June 30, 1963

(NASA Contract NASw-104) with 9 Amendments

Technical Management: National Aeronautics & Space Administration, Lewis Research Center
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OTS - cover

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FOREWORD

This report describes the research activity carried out in fulfillment of contract NASw-104 as modified by Amendments 1 through 9, during the period from January 1 through June 30, 1963.

ABSTRACT

During the six-month period covered by this report emittance measurements were made and the long term endurance tests were concluded. The total hemispherical emittance rig has been returned to service and the quality of temperature measurement has been investigated. An analysis of slurry coating procedures is being conducted.

Total hemispherical emittance measurements were made and are reported for AISI-310 stainless steel, tantalum, and coatings of crystalline boron, nickel-chrome spinel, calcium titanate, and iron titanate. All coating materials were plasma-arc sprayed onto columbium - 1 per cent zirconium tubes. Emittance values of 0.87 or above were obtained with coatings of nickel-chrome spinel, calcium titanate, and iron titanate. An endurance test conducted on a calcium-titanate coated specimen resulted in lower emittance values than anticipated but these values are not believed to be characteristic of this coating. Endurance tests were not completed for nickel-chrome spinel or iron titanate coatings.

An aluminum-phosphate bonded mixture of nickel-chrome spinel and silicon dioxide on a SNAP-8 finned-tube radiator segment completed 15,000 hours of endurance testing. Flame-sprayed coatings of titania on SNAP-8 and Sunflower I sections completed 14,037 and 13,755 hours respectively. A fourth rig containing a SNAP-8 section with an aluminum-phosphate bonded mixture of silicon carbide and silicon dioxide completed 12,781 hours of testing. All tests have been terminated and the coated segments are being maintained in vacuum at ambient temperatures pending post-test analysis.

The difficulties arising from the volatilization of manganese oxide in the total hemispherical emittance rig have been resolved and the rig was returned to service.

The quality of black-body hole configurations was re-evaluated and results substantiated previous analytical work. The new evaluation included the investigation of the effects of specimen misalignment and variations in chamber geometry. In conjunction with these investigations it has been found that, on the average, optical pyrometer temperature indications are 6°F higher than thermocouple indications. This problem is under investigation.

An investigation of Alkaphos-bonded coating procedures was conducted with attention being directed toward the bonding of silicon carbide. Nineteen tests were conducted using various surface preparation, drying, and curing procedures as well as various slurry compositions. The best procedure found to date requires 20 hours of air drying followed by oven curing at 200°F for 2 hours, 250°F for 2 hours, 300°F for 2 hours, and 400°F for 2 hours.

Author

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I. EMITTANCE MEASUREMENTS

The total hemispherical emittance of six materials was measured These materials were AISI-310 stainless steel, tantalum, and coatings of crystalline boron, nickel-chrome spinel, calcium titanate, and iron titanate

A. AISI-310 Stainless Steel

An AISI-310 stainless steel tube in the as-received condition was tested in the total hemispherical emittance rig in conjunction with the investigation of temperature measurement discrepancies discussed in section IV of this report. Emittance values were obtained between 1000 and 2000°F and appear in Table I and Figure 1. As shown, during initial heating the emittance was relatively constant at 0.27, but during the second run was lower. Similar drops in emittance have been experienced many times and have been attributed to surface clean-up resulting from exposure to elevated temperatures and reduced pressures.

TABLE I

Total Hemispherical Emittance

As Received, Uncoated AISI 310 Stainless Steel

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	ϵ th	Optical Pyrometer Temp. (°F)	ϵ th
1	0.2	4.4×10^{-7}	1000	0.264		
	0.6	3.6×10^{-7}	1202	0.276		
	0.8	3.4×10^{-7}	1401	0.285		
	1.2	9.2×10^{-7}	1600	0.289	1622	0.277
	1.6	2.0×10^{-6}	1800	0.279	1832	0.264
	1.9	2.0×10^{-6}	2000	0.287	2039	0.269
	Rig Opened; Specimen Removed and Later Reinstalled					
2	0.2	1.1×10^{-6}	999	0.211		
	0.5	1.1×10^{-6}	1333	0.246		
	0.8	1.1×10^{-6}	1500	0.258	1521	0.244

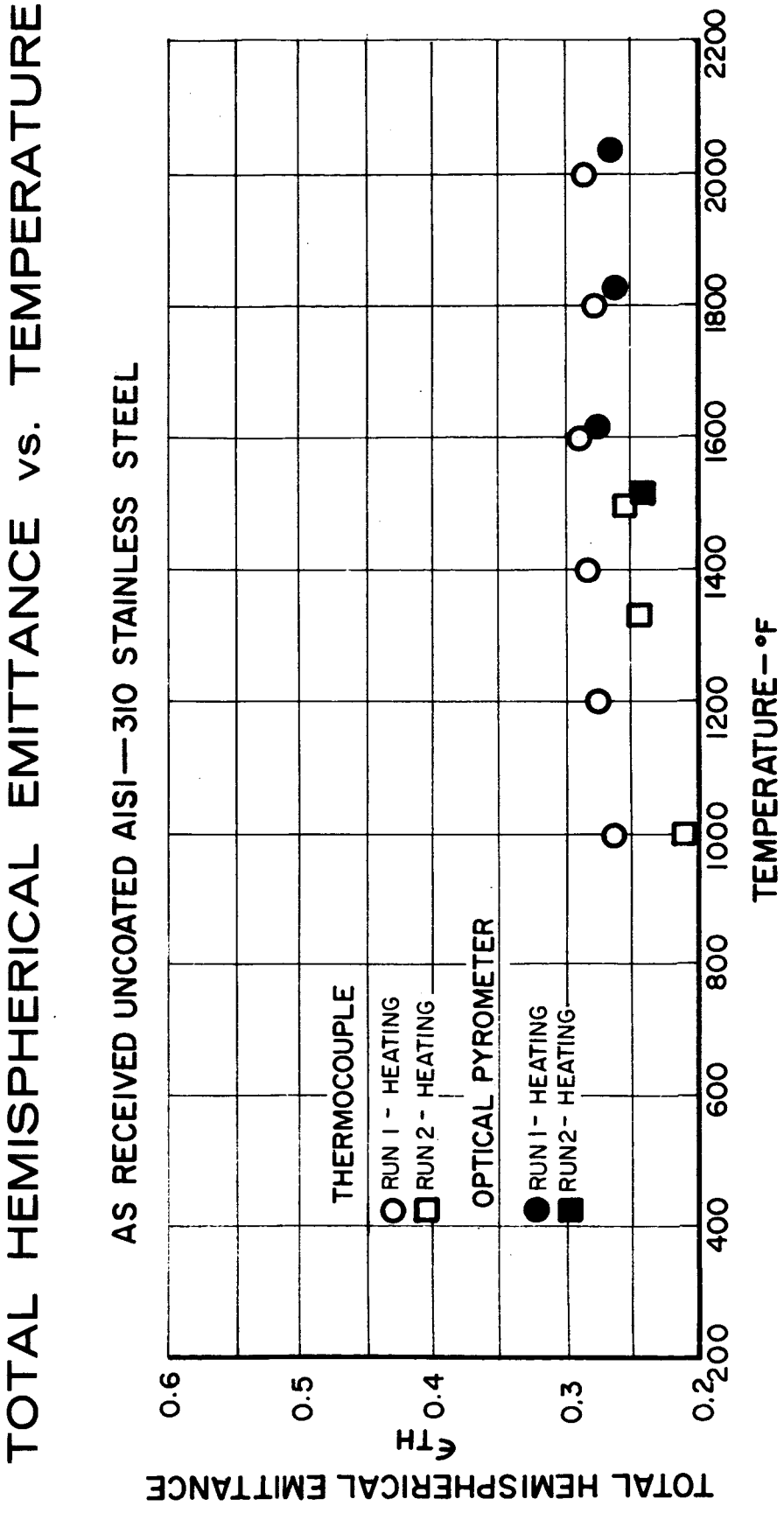
TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
AS RECEIVED UNCOATED AISI—310 STAINLESS STEEL
THERMOCOUPLE
RUN 1 - HEATING
RUN 2 - HEATING
OPTICAL PYROMETER
RUN 1 - HEATING
RUN2 - HEATING
TOTAL HEMISPHERICAL EMITTANCE
ϵ_{TH}
0.6
0.5
0.4
0.3
0.2
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE—°F

Figure 1

B. Tantalum

Five emittance runs were made with two tantalum tubes in conjunction with the investigation of temperature measurement discrepancies. Four runs were made with the first tube in the total hemispherical emittance rig and the final run was made with the second tube in the short term endurance rig. As shown in Table II and Figure 2, the data fall along two general curves with data from the first three runs of the first specimen comprising the lower curve and that from the last run of the first specimen and the single run of the second specimen comprising the upper curve. The lower curve indicates a gradual increase in emittance from 0.14 at 900°F to 0.20 at 2200°F whereas the upper curve increases from 0.17 at 1000°F to 0.25 at 2200°F. It is not known why the emittance should have increased on the final run with the first specimen nor why the initial run of the second should have been higher than that of the first. The specimens appeared similar and no change in their appearance resulted from testing. Both of these tubes had been previously tested so that additional changes were not expected.

TABLE II

Total Hemispherical Emittance

Uncoated Polished Tantalum

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	ϵ th	Optical Pyrometer Temp. (°F)	ϵ th
1	0.4	1.0×10^{-6}	1499	0.164	1516	0.158
	0.8	1.1×10^{-6}	2201	0.200	2221	0.194
	1.8	1.1×10^{-6}	2200	0.197	2226	0.190
2	2.0	1.0×10^{-6}	904	0.139		
	2.4	1.0×10^{-6}	999	0.140		
	2.6	8.7×10^{-7}	1100	0.142		
	2.9	7.5×10^{-7}	1201	0.144		
	3.2	7.3×10^{-7}	1300	0.150		
		Heating Current Off; Vacuum Maintained				
3	3.5	9.8×10^{-7}	1000	0.139		
	3.8	9.1×10^{-7}	1200	0.145		
	3.9	1.0×10^{-6}	1400	0.154		
	4.5	1.0×10^{-6}	1600	0.165	1608	0.162
	4.8	1.0×10^{-6}	1801	0.175	1817	0.170
	5.1	1.1×10^{-6}	2000	0.183	2020	0.178
	5.3	1.4×10^{-6}	2199	0.195	2226	0.187
	5.4	1.0×10^{-6}	1503	0.147	1516	0.144
	5.5	9.3×10^{-7}	1000	0.133		
		Rig Opened; Specimen Removed and I ater Reinstalled				
4	0.1	4.1×10^{-7}	1000	0.167		
	0.2	5.8×10^{-7}	1200	0.169		
	0.3	1.9×10^{-6}	1400	0.180		
	0.5	6.6×10^{-7}	1600	0.207	1592	0.210
	0.6	4.9×10^{-7}	1800	0.230	1789	0.234
	1.0	4.0×10^{-7}	2000	0.256	1994	0.259
	1.1	5.1×10^{-7}	2200	0.256	2215	0.250
	1.2	4.8×10^{-7}	2150	0.237	2164	0.232
	1.3	3.4×10^{-7}	1850	0.218	1859	0.215
	1.4	3.4×10^{-7}	1550	0.203	1559	0.200
	1.5	3.0×10^{-7}	1249	0.185		

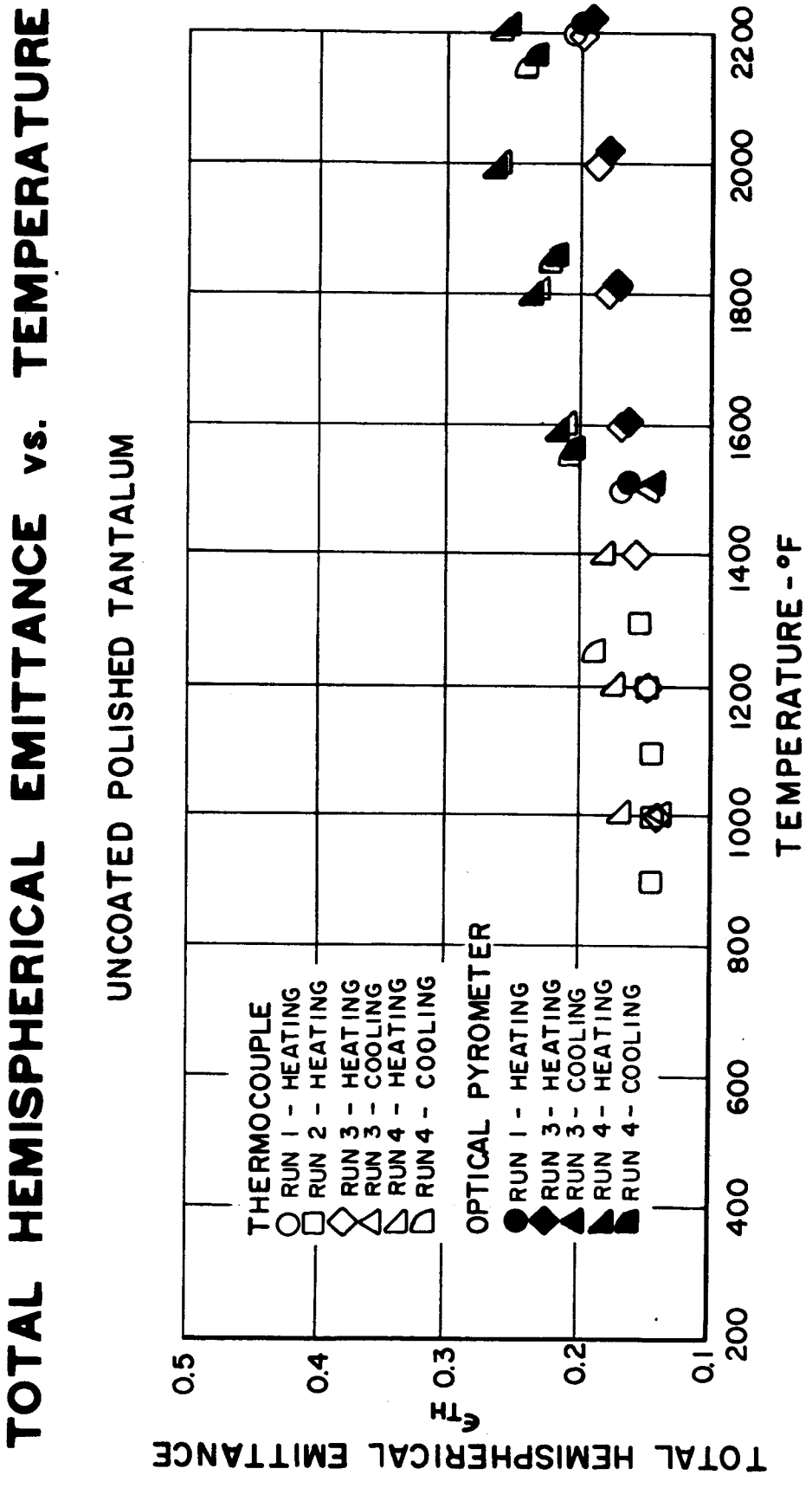
TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
UNCOATED POLISHED TANTALUM
THERMOCOUPLE
RUN 1 - HEATING
RUN 2 - HEATING
RUN 3 - HEATING
RUN 3 - COOLING
RUN 4 - HEATING
RUN 4 - COOLING
OPTICAL PYROMETER
RUN 1 - HEATING
RUN 3 - HEATING
RUN 3 - COOLING
RUN 4 - HEATING
RUN 4 - COOLING
TOTAL HEMISPHERICAL EMITTANCE
ϵ_{TH}
0.5
0.4
0.3
0.2
0.1
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE - °F

Figure 2

C. Crystalline Boron

A coating of crystalline boron less than 1 mil thick was plasma-arc sprayed onto a columbium-1 per cent zirconium tube. A thicker coating could not be obtained since only the first layer of powder applied to the substrate would adhere. The powder used had particles with diameters ranging from 62 to 74 microns. The coating was grey, fairly hard, and had a matte texture smoother than that of 320 grit emery cloth. The coating-substrate bond strength was excellent.

Total hemispherical emittance measurements were made between 300 and 2200°F and data are presented in Table III and Figure 3. The emittance increased from 0.65 at 300°F to 0.74 at 1300°F where it remained up to 1700°F. During the second heating cycle the emittance obtained between 1500 and 1700°F was slightly lower than previously and with further heating the emittance started decreasing at 1900°F so that at 2200°F it was 0.70. During cooling the emittance remained at or below 0.70. The lowest emittance data previously reported (Technical Report PWA-2206) for crystalline boron was slightly higher than the value obtained with this specimen at 1400°F. It is possible that the lower values resulted from the thinness of the coating. No visible changes in the coating occurred as a result of testing.

TABLE III

Total Hemispherical Emittance

Coating: Crystalline Boron - Plasma Arc Sprayed (< 1-mil)
Substrate: Columbium - 1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	ϵ th	Optical Pyrometer Temp. (°F)	ϵ th
1	0.6	4.6×10^{-7}	300	0.651		
	0.7	4.0×10^{-7}	500	0.685		
	1.0	4.4×10^{-7}	700	0.691		
	1.2	4.3×10^{-7}	900	0.705		
	1.3	8.0×10^{-7}	1000	0.710		
	1.4	5.2×10^{-7}	1100	0.721		
	1.5	5.6×10^{-7}	1200	0.728		
	1.6	4.1×10^{-7}	1300	0.736		
	1.7	1.2×10^{-6}	1400	0.740		
	1.8	6.6×10^{-7}	1500	0.747	1505	0.740
	2.3	6.2×10^{-7}	1600	0.752	1611	0.736
	2.7	1.8×10^{-6}	1700	0.757	1711	0.742
		Heating Current Off; Vacuum Maintained				
2	3.0	5.0×10^{-7}	1500	0.727	1514	0.707
	3.1	5.0×10^{-7}	1600	0.734	1611	0.718
	3.3	4.0×10^{-7}	1700	0.738	1710	0.724
	3.7	5.0×10^{-7}	1800	0.745	1811	0.731
	3.9	1.0×10^{-6}	1900	0.742	1913	0.725
	4.5	6.8×10^{-7}	2000	0.730	2019	0.707
	4.8	1.3×10^{-6}	2200	0.699	2225	0.673
	4.9	1.0×10^{-6}	2150	0.677	2169	0.657
	5.0	5.0×10^{-7}	1850	0.622	1859	0.612
	5.2	2.8×10^{-7}	1550	0.643	1562	0.643
	5.4	1.6×10^{-7}	1250	0.621		

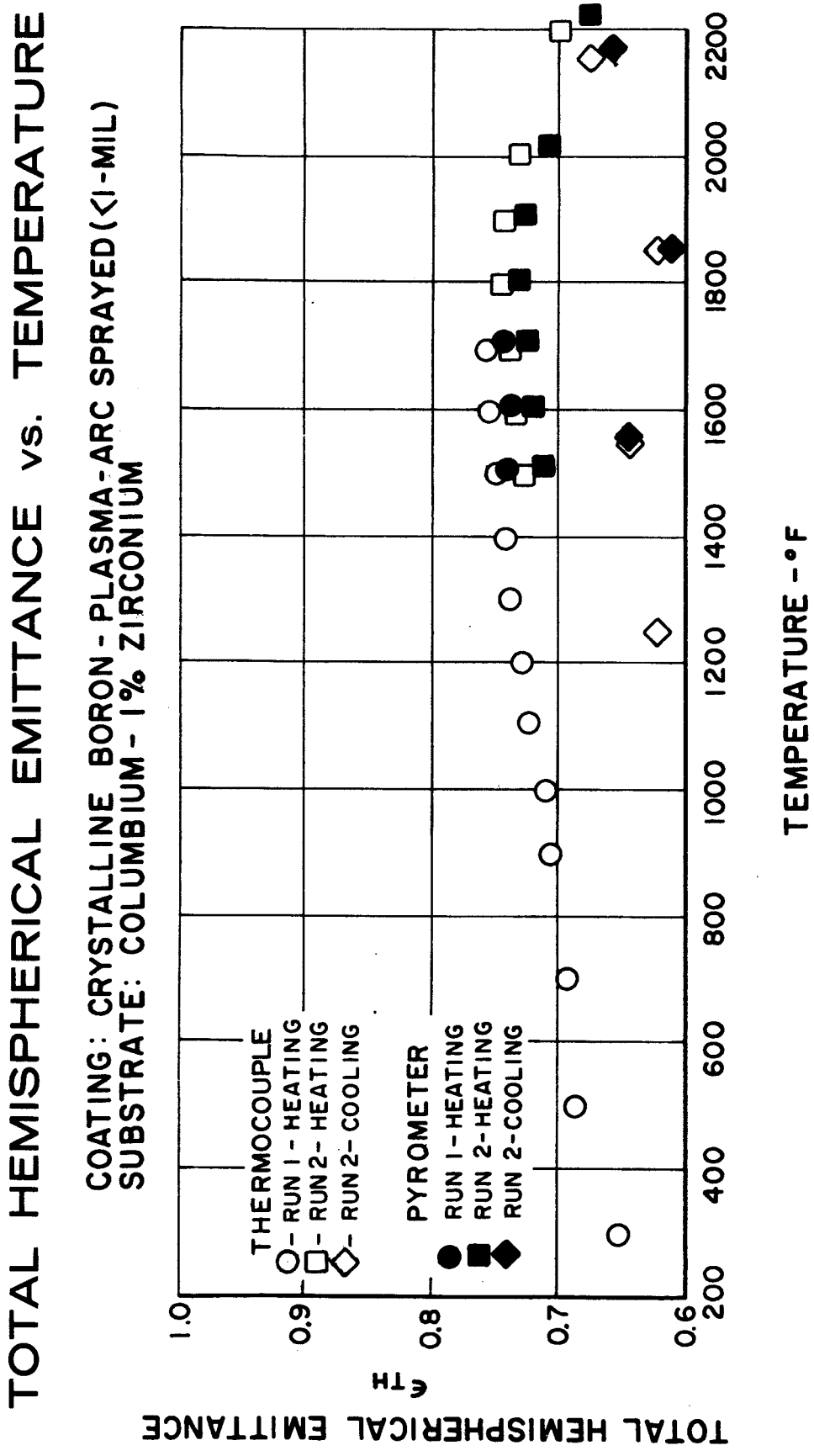

Figure 3
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D. Nickel-Chrome Spinel

Nickel-chrome spinel ($NiO \cdot Cr_2O_3$) was made at Pratt & Whitney Aircraft by heating a stoichiometric mixture of nickel oxide and chrome oxide at 2500°F for 10 hours. X-ray diffraction analysis determined that the resulting material was 70 per cent $NiO \cdot Cr_2O_3$, 25 per cent Cr_2O_3, and 5 per cent NiO.

A 2-mil thick coating of this material was plasma-arc sprayed onto a columbium- 1 per cent zirconium tube. As was the case with crystalline boron, difficulty was encountered in obtaining a thick coating. The coating was black, soft, and had a matte texture similar to that of 240 grit emery cloth. The coating-substrate bond strength was poor.

The total hemispherical emittance was measured between 300 and 2200°F and results appear in Table IV and Figure 4. The emittance increased from about 0.78 at 300°F to 0.87 at 900°F. It remained at 0.87 between 900°F and 2100°F. At 2200°F a bright spot appeared at the bottom of the tube and more power was required to maintain the temperature level. During cooling the emittance data retraced that obtained during heating.

After the specimen was removed from the rig it was found that the lower part of the specimen which had overheated during testing had turned white while the remainder of the specimen remained black. The black coating was fairly hard and now had a fair coating-substrate bond strength. A shiny metal coating was found on the interior of the chamber and on the instrumentation flange. It appears that there was some defect or impurity in the coating at the lower end of the specimen since this is the only place where an observable change in the coating occurred.

The emittance data presented here confirms that the line shown in Figure 133 of Technical Report PWA-2206 for aluminum phosphate bonded nickel-chrome spinel is the most probable emittance level of this material. A thicker coating should be tested to determine if the initial emittance increase was a result of transparency of the coating at the longer wavelengths.

TABLE IV

Total Hemispherical Emittance

Coating: Nickel Chrome Spinel - Plasma Arc Sprayed (2-mil)
Substrate: Columbium - 1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	ϵ th	Optical Pyrometer Temp. (°F)	ϵ th
1	0.7	1.5×10^{-7}	300	0.786		
	0.9	1.2×10^{-7}	500	0.839		
	1.2	1.2×10^{-7}	700	0.865		
	1.4	2.5×10^{-7}	900	0.874		
	1.5	2.8×10^{-7}	1000	0.873		
	2.0	3.6×10^{-7}	1100	0.878		
	2.1	4.9×10^{-7}	1200	0.876		
	2.3	4.8×10^{-7}	1300	0.878		
	2.6	4.2×10^{-7}	1400	0.879		
	3.6	4.2×10^{-7}	1500	0.871	1500	0.871
	3.8	5.4×10^{-7}	1600	0.870	1601	0.869
	4.1	1.0×10^{-7}	1700	0.867	1701	0.865
	4.3	1.4×10^{-6}	1800	0.866	1807	0.855
	4.5	1.4×10^{-6}	1900	0.868	1906	0.859
	4.7	1.6×10^{-6}	2000	0.867	2003	0.863
	4.9	1.6×10^{-6}	2100	0.878	2097	0.881
	5.1	2.2×10^{-6}	2200	0.878	2189	0.893
	5.2	8.4×10^{-6}	2150	0.883	2148	0.885
	5.4	1.0×10^{-6}	1850	0.880	1861	0.864
	5.6	6.2×10^{-7}	1550	0.872		

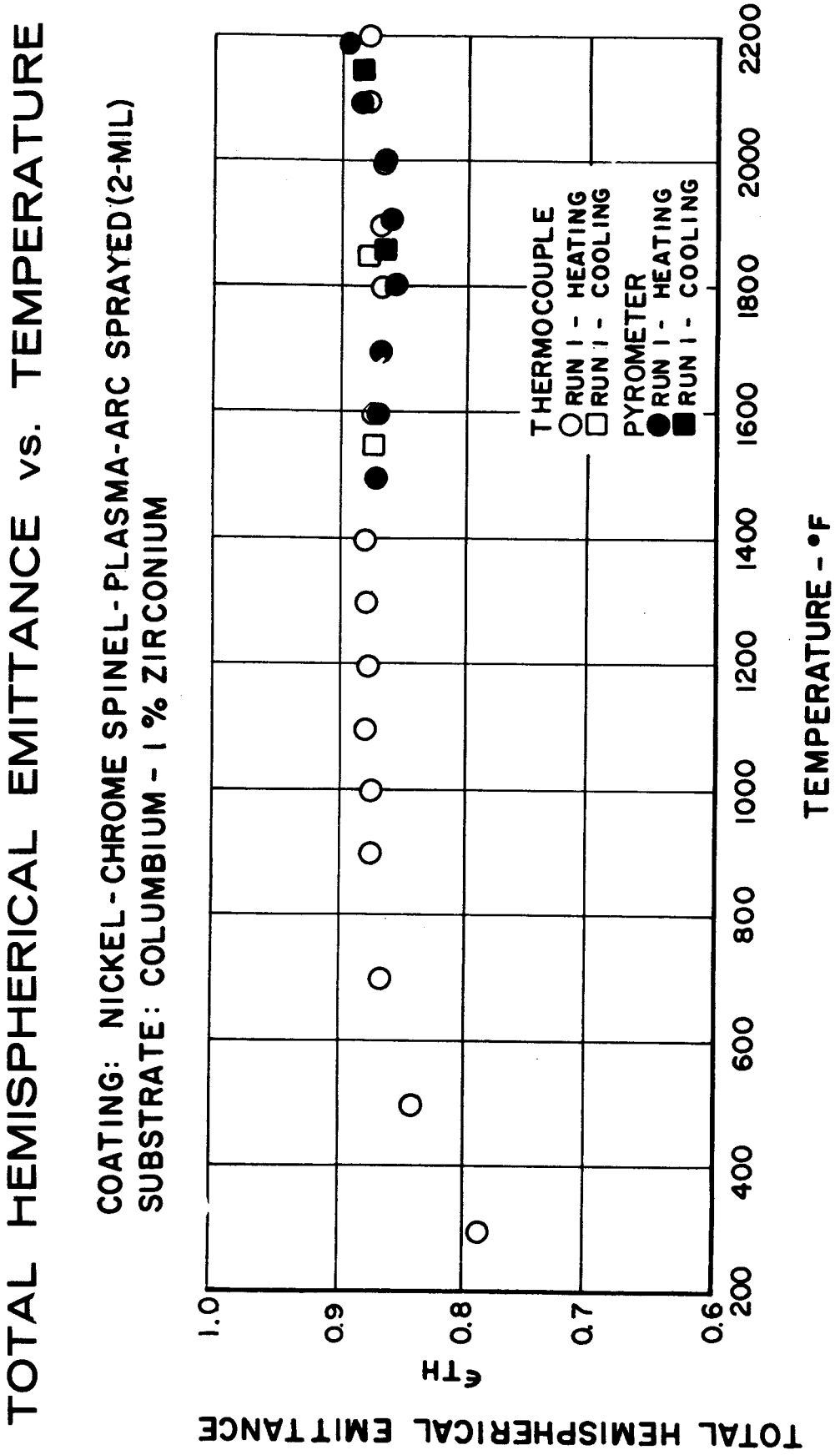

Figure 4
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E. Calcium Titanate

High-purity calcium titanate ($CaO \cdot TiO_2$) obtained from the Titanium Division of the National Lead Company was plasma-arc sprayed onto columbium-1 per cent zirconium tubes. The resulting coatings were 5 mils thick, hard, and had a matte texture similar to that of 240 grit emery cloth. The coating-substrate bond strengths were good. One of the specimens was tested in the total hemispherical emittance rig and one in the short term endurance rig.

Total Hemispherical Emittance Rig - The specimen tested in the total hemispherical emittance rig had a light gray coating. Emittance measurements were made between 300°F and 1600°F and results are presented in Table V and Figure 5. The emittance of the coating increased from 0.78 at 300°F to about 0.90 at 1300°F and remained at that level to 1600°F at which time the test was terminated. These values agree with those anticipated for $CaO \cdot TiO_2$ and $SrO \cdot TiO_2$.

When the specimen first reached 1000°F it was observed that the color of the chamber walls changed from gray to a brilliant blue. As may be seen in Table V, the pressure level rose one decade at 900°F, remained at that level at 1000°F, and then returned to its original level at 1100°F. The pressure remained in the 10^{-7} mm Hg range until temperatures in excess of 1300°F were attained and then it once again rose to the 10^{-6} mm Hg range. The chamber pressure was low enough during the period when the color changed that it was not believed that excessive volatilization had taken place and termination of the test was not warranted. Also, no extra power was required to maintain the specimen temperature as would be required if excessive volatilization had occurred. Finally, it would not be expected that condensate from a calcium-titanate coating would be blue. When the vacuum chamber was opened no foreign material was found on the instrumentation flange although the chamber walls were still blue. The cause of this blue coating has not been determined at the present time. There were no changes in the characteristics of the coating as a result of testing

Short Term Endurance Rig

Numerous measurements of the emittance of calcium titanate and strontium titanate have established that the emittances of these

materials gradually increase until a value of about 0.90 is attained at a temperature between 1300 and 1400°F. This value is retained during cooling. Further, the emittance of one calcium titanate specimen increased slightly when the specimen was endurance tested for 17 hours at 1450°F (see Technical Report PWA-2206, page 112). It was therefore decided to conduct an endurance test to determine if the total emittance of calcium-titanate would rise to the 0.9 level at temperatures below 1400°F.

The coating contained blue and white particles on a light gray background and was tested between 300 and 1500°F. Results appear in Table VI and Figures 6 through 8. The total hemispherical emittance of the coating was measured as the specimen was heated from 300 to 1000°F (see Figure 6). A 190-hour endurance test was conducted at 1000°F. As shown in Figure 7, the emittance rose slightly during the first 50 hours from 0.84 to 0.86, and then remained relatively constant throughout the last 140 hours. The specimen was then cooled to 600°F and then heated to 1100°F (see Figure 6). Successive endurance runs were made at 1100°F, 1200°F, 1300°F, and 1400°F with no change in the emittance noted. Most of the endurance tests were run for one day but the test at 1300°F encompassed a weekend and was therefore somewhat longer. At 1500°F the emittance began to decrease and after 28 hours it had dropped from 0.86 to 0.72 (see Figure 8). After 28 hours at 1500°F the temperature of the specimen was decreased and the emittance remained at 0.72, thus indicating that a permanent change in the coating had occurred. The thermocouple data appears to be reliable since at the end of the endurance test at 1500°F thermocouple and optical pyrometer indications were in good agreement. After testing the coating was uniformly gray. Similar color changes with calcium titanate specimens have occurred previously (see Technical Report PWA-2206, page 115).

The results from this test were not as anticipated since the emittance of the specimen never attained 0.90 even at 1400 or 1500°F. Further, it has been shown (Technical Report PWA-2206, Table 144) that calcium titanate is stable for up to 400 hours at 1450°F. Since the performance of this specimen was not similar to that of other calcium titanate specimens, no conclusions may be drawn concerning low-temperature enduring of calcium titanate coated specimens until further testing is conducted.

TABLE V

Total Hemispherical Emittance

Coating: Calcium Titanate - Plasma-Arc Sprayed (5-mil)
Substrate: Columbium - 1% Zirconium

First Specimen

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	ϵ th	Optical Pyrometer Temp. (°F)	ϵ th
1	0.8	3.7×10^{-7}	300	0.778		
	2.1	3.1×10^{-7}	500	0.814		
	2.5	3.2×10^{-7}	700	0.830		
	3.1	1.0×10^{-6}	900	0.850		
	3.6	1.3×10^{-6}	1000	0.856		
	3.8	5.0×10^{-7}	1100	0.861		
	4.1	4.7×10^{-7}	1198	0.884		
	4.4	5.7×10^{-7}	1300	0.897		
	4.7	1.4×10^{-6}	1400	0.900		
	4.8	1.2×10^{-6}	1500	0.904	1511	0.884
	5.0	7.9×10^{-7}	1252	0.880		
2	5.2	1.6×10^{-6}	1601	0.895	1608	0.883

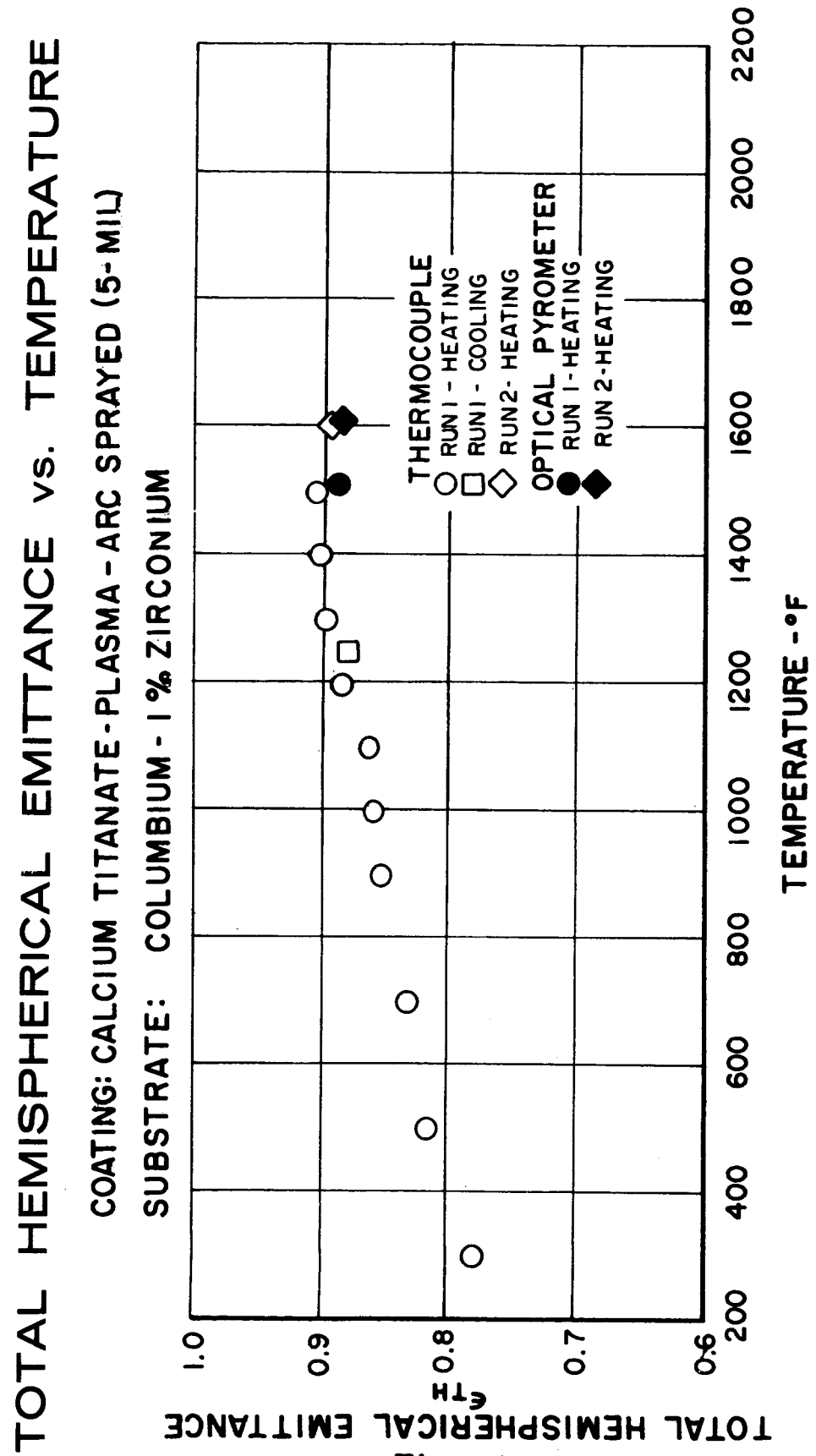

Figure 5
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TABLE VI

Total Hemispherical Emittance
Coating: Calcium Titanate - Plasma-Arc Sprayed (5-Mil)
Substrate: Columbium-1% Zirconium
Second Specimen

Run Number	Elapsed Time (Hrs.)	Endurance Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp.(°F)	Thermocouple ϵ th	Thermocouple Avg. ϵ th	Optical Pyrometer Temp(°F)	Optical Pyrometer ϵ th
1	0.2		1.6×10^{-8}	300	0.875			
	0.6		3.0×10^{-7}	500	0.876			
	1.0		7.0×10^{-7}	700	0.843			
	1.3		1.7×10^{-6}	900	0.838			
	1.7	0.0	9.2×10^{-7}	1000	0.834			
	2.9	1.2	2.2×10^{-7}	1000	0.835	0.839		
	4.3	2.6	1.3×10^{-7}	1000	0.839			
	6.0	4.3	6.5×10^{-8}	1000	0.846			
	23.5	21.8	2.0×10^{-8}	1000	0.858	0.859		
	30.7	29.0	2.2×10^{-8}	1000	0.860			
	47.6	45.9	1.3×10^{-8}	1000	0.855	0.856		
	52.8	51.1	1.4×10^{-8}	1000	0.857			
	54.6	52.9	1.4×10^{-8}	999	0.859			
	71.3	69.6	1.1×10^{-8}	999	0.853	0.855		
	76.1	74.4	1.1×10^{-8}	999	0.853			
	95.3	93.6	1.1×10^{-8}	999	0.856	0.856		
	102.6	100.9	1.1×10^{-8}	999	0.857			
	168.0	166.3	7.0×10^{-9}	999	0.861	0.859		
	175.0	173.3	7.0×10^{-9}	1001	0.857			
	191.4	189.7	6.0×10^{-9}	1001	0.853			
	191.8		5.0×10^{-9}	799	0.867			
	192.2		4.0×10^{-9}	601	0.870			
2	192.5		5.0×10^{-9}	800	0.864			
	192.8		5.5×10^{-9}	1000	0.855			
	193.1	0.0	1.5×10^{-8}	1100	0.856			
	194.1	1.0	1.8×10^{-8}	1101	0.855			
	195.0	1.9	1.9×10^{-8}	1099	0.858	0.857		
	198.8	5.7	1.9×10^{-8}	1100	0.858			
	215.4	22.3	1.5×10^{-8}	1100	0.858			
	216.7	0.0	6.0×10^{-8}	1199	0.863			
	218.9	2.2	5.1×10^{-8}	1200	0.862			
	222.5	5.8	4.7×10^{-8}	1200	0.862	0.863		
	239.5	22.8	3.5×10^{-8}	1200	0.866			
	240.8	0.0	1.7×10^{-7}	1300	0.866			
	242.0	1.2	2.0×10^{-7}	1300	0.867			
	246.7	5.9	1.3×10^{-7}	1300	0.867			
	263.4	22.6	6.2×10^{-8}	1300	0.865			
	267.0	26.2	6.2×10^{-8}	1300	0.865			
	270.6	29.8	6.0×10^{-8}	1300	0.866	0.866		
	335.8	95.0	2.6×10^{-8}	1300	0.865			
	339.7	98.9	3.1×10^{-8}	1300	0.866			
	342.5	101.7	3.0×10^{-8}	1300	0.865			
	359.1	118.3	2.2×10^{-8}	1300	0.866			
	359.9	0.0	6.8×10^{-8}	1400	0.865			
	363.0	3.1	9.0×10^{-8}	1400	0.865	.865		
	366.9	7.0	9.1×10^{-8}	1400	0.863			
	383.3	23.4	6.2×10^{-8}	1400	0.858			
	383.8	0.0	4.8×10^{-7}	1500	0.858			
	388.5	4.7	1.9×10^{-7}	1500	0.833			
	390.9	7.1	1.6×10^{-7}	1500	0.833			
	407.7	23.9	4.0×10^{-8}	1500	0.730			
	411.5	27.7	3.2×10^{-8}	1500	0.722		1499	0.723
	412.7		1.7×10^{-8}	1400	0.720			
	413.0		1.2×10^{-8}	1300	0.719			
	413.2		9.5×10^{-9}	1200	0.720			
	413.5		7.7×10^{-9}	1100	0.724			
	413.8		6.0×10^{-9}	1000	0.535			

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

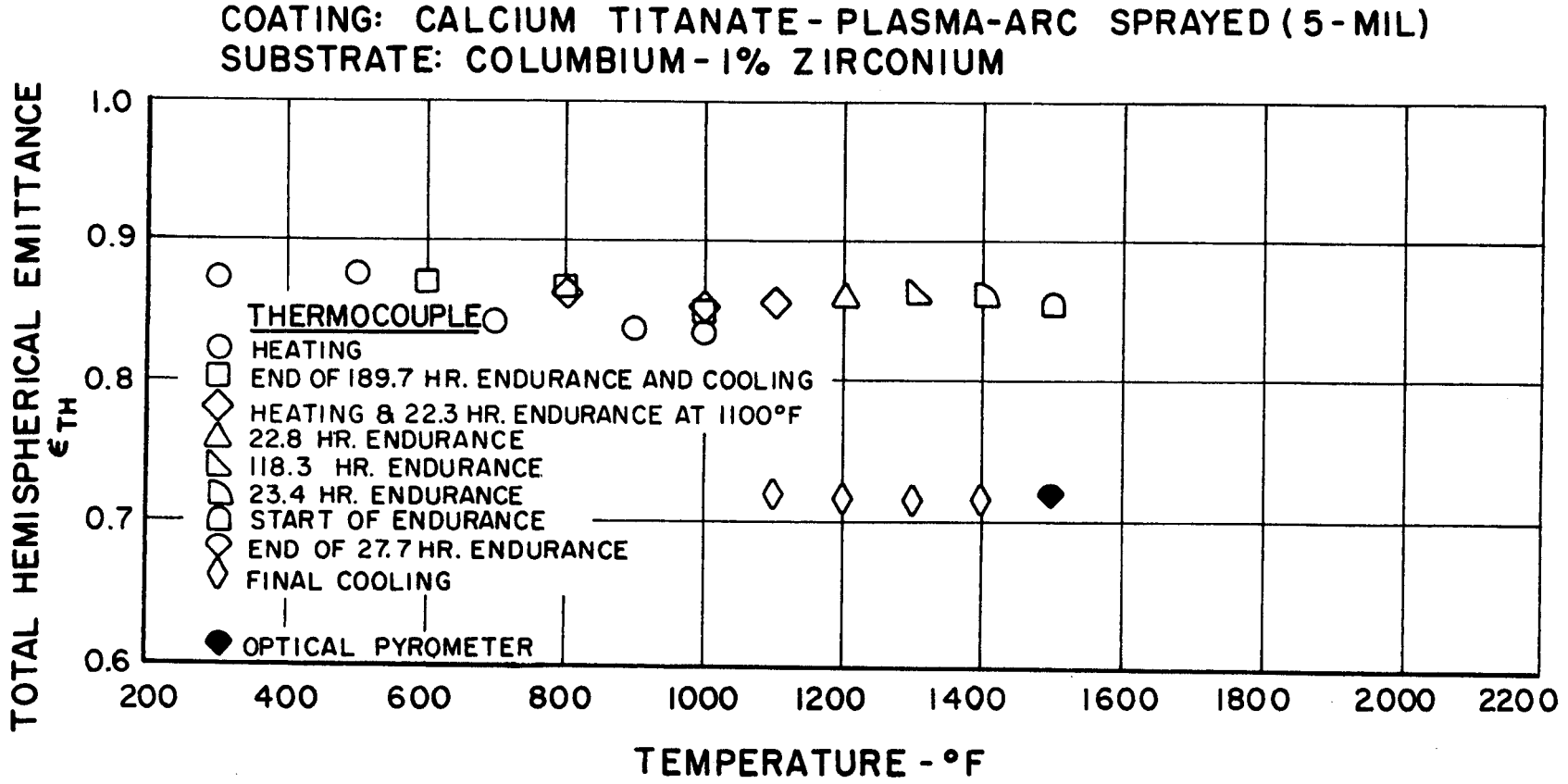

Figure 9
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PWA-2255

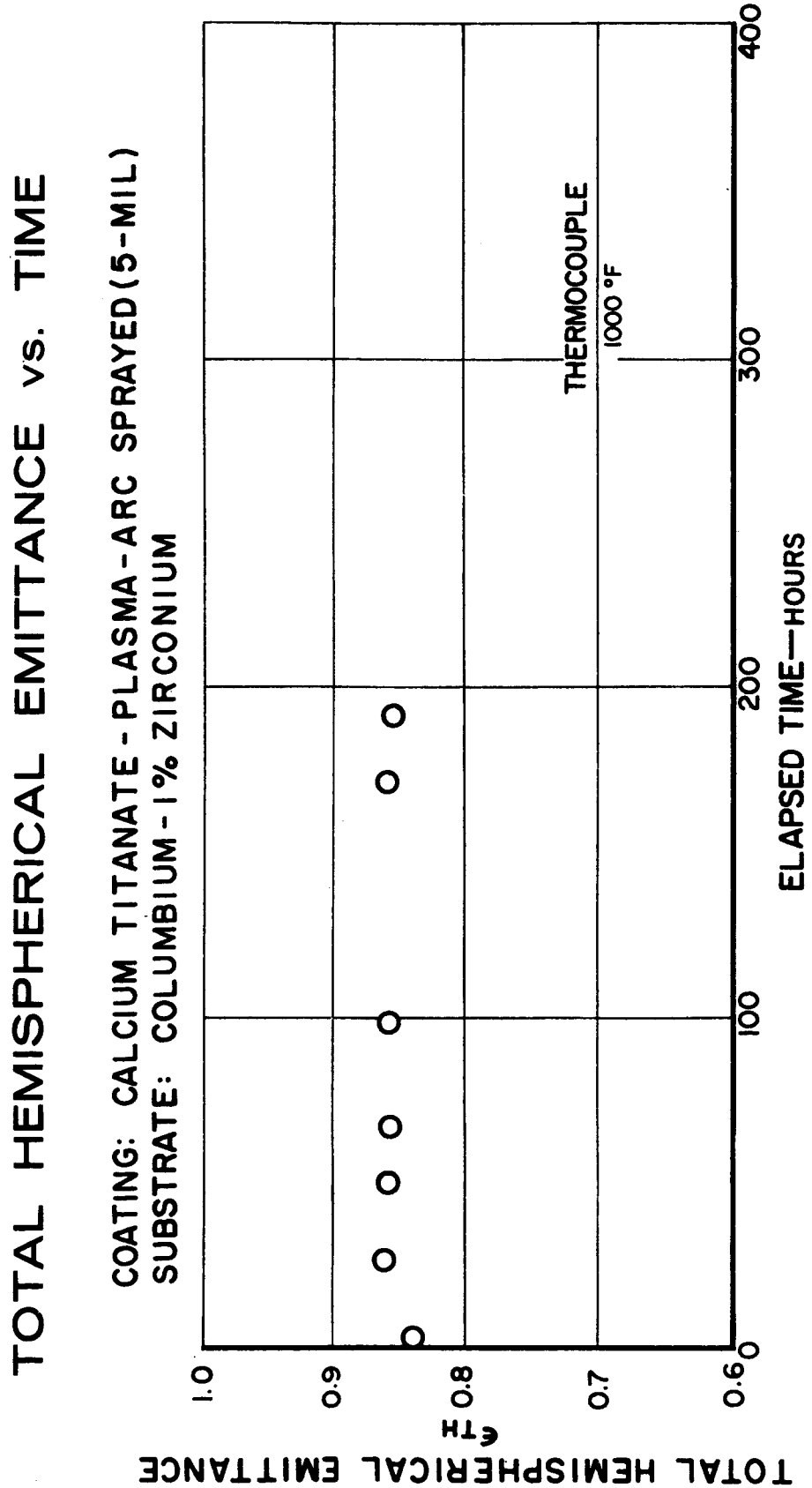

TOTAL HEMISPHERICAL EMITTANCE vs. TIME
COATING: CALCIUM TITANATE - PLASMA-ARC SPRAYED (5-MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
1000 °F
TOTAL HEMISPHERICAL EMITTANCE
εTH
1.0
0.9
0.8
0.7
0.6
0
100
200
300
400
ELAPSED TIME—HOURS

Figure 7

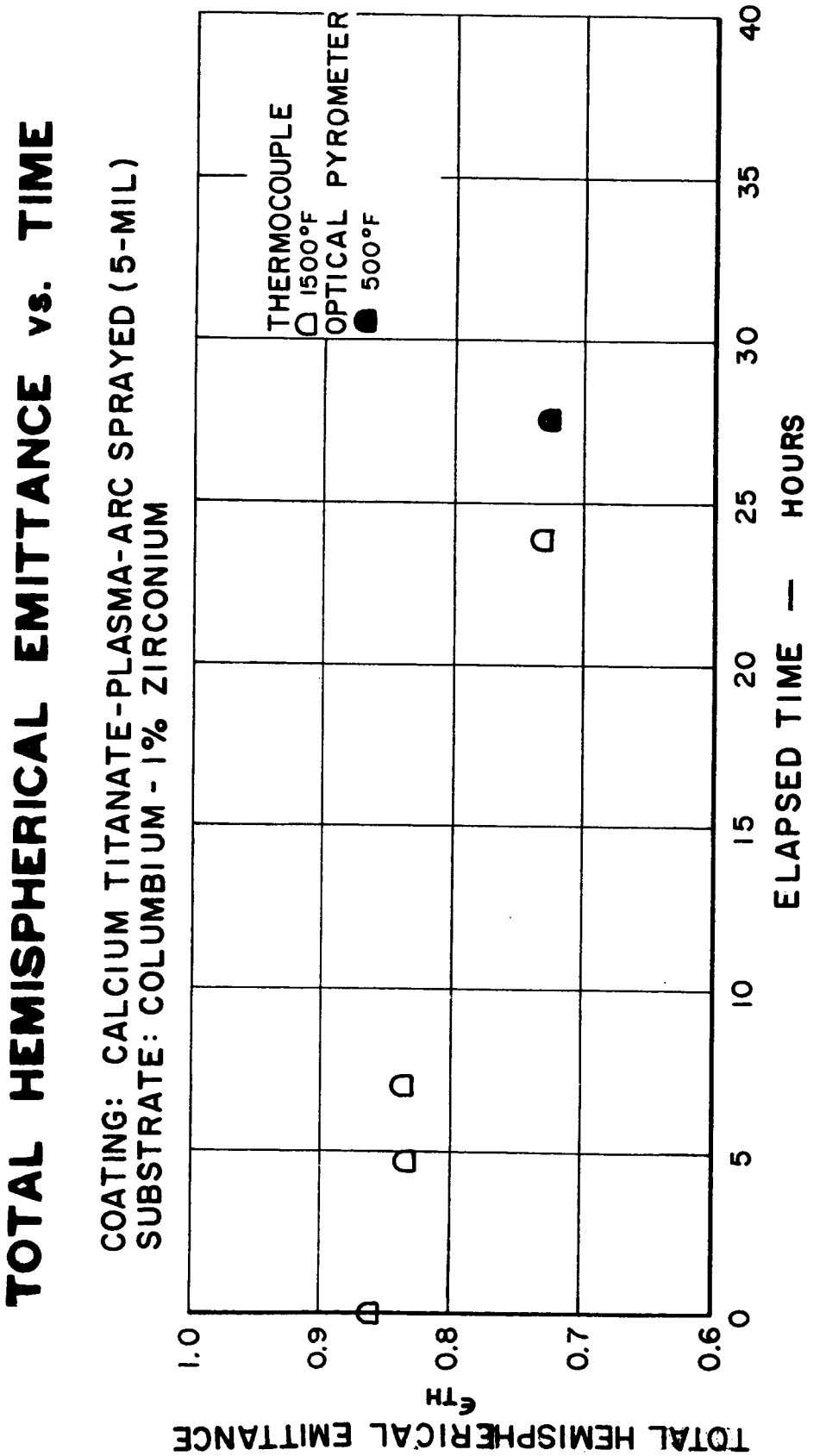


Figure 8
Page 20

F. Iron Titanate

Iron-titanium-oxide (FCT-11) was obtained from the Continental Coatings Corporation and was determined by X-ray diffraction to be Fe_2TiO_5. The iron titanate was plasma-arc sprayed onto columbium-1 per cent zirconium and the resultant coatings were dark gray, hard, and had a matte texture similar to that of 320 grit emery cloth. The coating-substrate bond strength was good. During this report period two specimens were tested, one with a coating 5 mils thick in the total emittance rig, and the other with a coating 4 mils thick in the short term endurance rig.

Total Hemispherical Emittance Rig - Emittance measurements were made only over the temperature range of 1000 to 2200°F in order to quickly determine whether or not the emittance level of the new iron-titanate coated specimens was comparable to that of similar specimens tested previously. If the emittance level was comparable then endurance testing at 1800°F would be warranted.

As can be seen in Table VII and Figure 9, the emittance level of this specimen was about 0.87 between 1000 and 2100°F which is a value close to that obtained previously. However, at 2200°F the coating started to volatilize and therefore reliable data could not be obtained during cooling. After the specimen was removed from the rig it was found that the chamber walls and instrumentation flange were coated but the characteristics of the coating on the specimen appeared unchanged. Although the coating volatilized at 2200°F, it was decided to endurance test a specimen at 1800°F since the coating was stable at least up to a temperature of 2000°F and since data obtained up to 2100°F were equivalent to that obtained previously. However, before this coating material is tested at temperatures above 2000°F a volatilization test will be run. The test will determine the highest temperature at which iron titanate can function in a 10^{-8} mm Hg vacuum as a space radiator coating.

Short Term Endurance Rig - The specimen with the 4-mil thick coating of iron-titanate was heated to 1800°F in the short term endurance rig. During heating the emittance varied between 0.86 and 0.87 (see Table VIII and Figure 10). After the specimen had

been at 1800°F with an emittance of 0.87 for 1.6 hours, however, a partial blockage of the cooling water caused overheating of the chamber walls and the instrumentation flange and the test was terminated. The specimen was a darker shade of gray after testing than before but no other changes were found. The investigation of the endurance characteristics of this material will be continued.

TABLE VII

Total Hemispherical Emittance

Coating: Iron Titanate - Plasma-Arc Sprayed (5-mil)
Substrate: Columbium - 1% Zirconium

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	ϵ th	Optical Pyrometer Temp. (°F)	ϵ th
1	0.2	9.1×10^{-7}	1000	0.872		
	0.5	5.5×10^{-7}	1100	0.872		
	0.7	4.6×10^{-7}	1199	0.874		
	1.0	4.4×10^{-7}	1300	0.872		
	1.3	3.3×10^{-7}	1401	0.871		
	1.7	2.2×10^{-7}	1501	0.871	1516	0.843
	2.9	2.5×10^{-7}	1600	0.866	1606	0.856
	3.3	2.4×10^{-7}	1700	0.869	1704	0.863
	3.5	2.6×10^{-7}	1800	0.872	1805	0.865
	4.1	2.7×10^{-7}	1903	0.869	1910	0.859
	4.5	4.8×10^{-7}	2001	0.870	2029	0.831
	4.7	1.1×10^{-6}	2099	0.886	2113	0.867
	4.9	8.9×10^{-6}	2180	0.911	2174	0.919

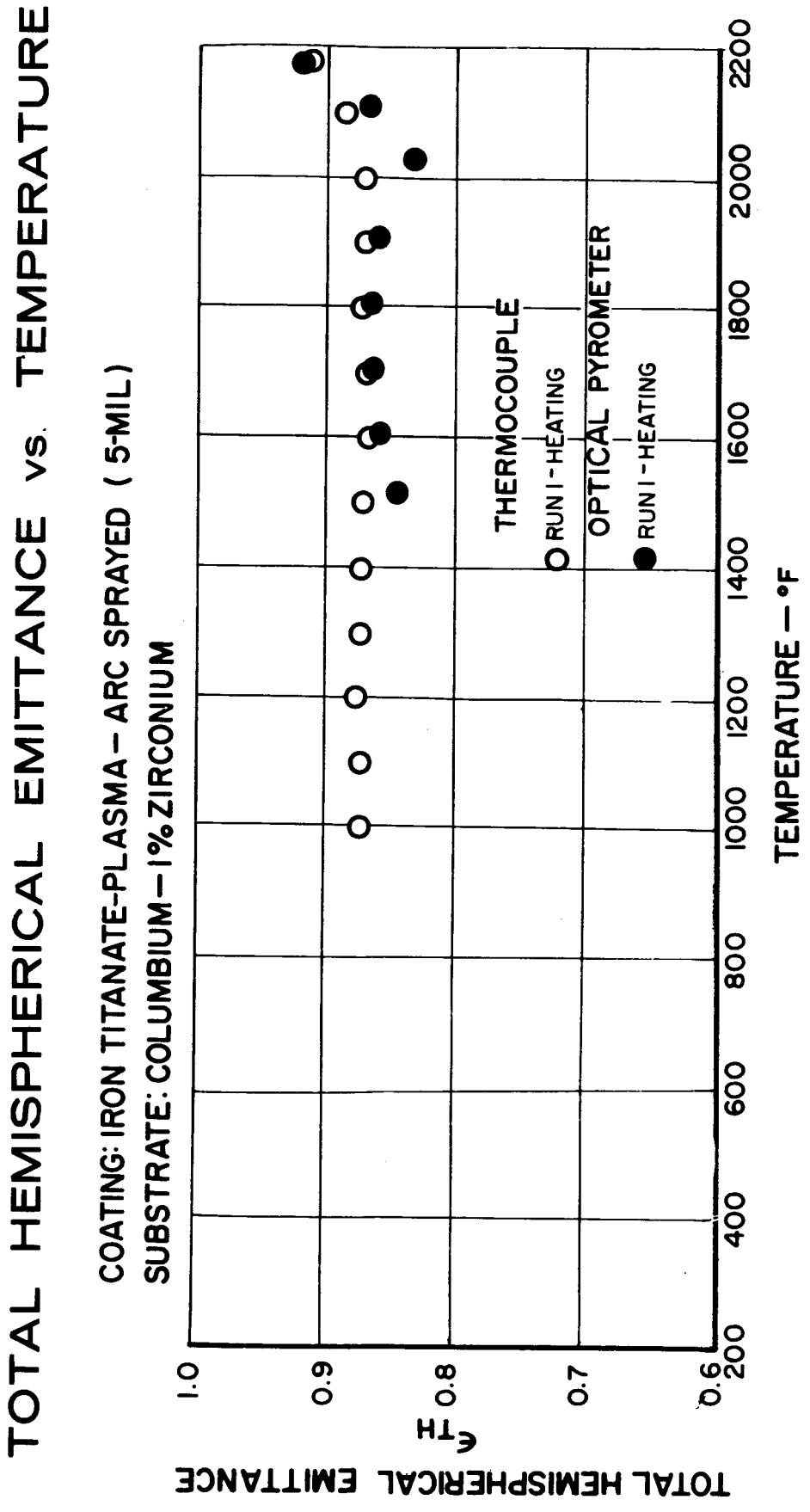


Figure 9
Page 24

TABLE VIII

Coating: Iron Titanate
Substrate: Columbium - 1% Zirconium

4-Mil Coating

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ th	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ th
1	0.2	1.7×10^{-6}	700	0.863		
	0.4	2.1×10^{-6}	800	0.865		
	0.6	2.1×10^{-6}	900	0.859		
	0.9	2.9×10^{-6}	1001	0.857		
	1.2	2.2×10^{-6}	1099	0.866		
	1.9	4.7×10^{-7}	1199	0.864		
	2.2	1.0×10^{-6}	1300	0.867		
	2.6	1.5×10^{-6}	1400	0.870		
	3.1	3.6×10^{-6}	1500	0.869	1502	0.866
	3.5	3.0×10^{-6}	1600	0.869	1601	0.868
	3.9	5.6×10^{-6}	1700	0.872	1700	0.872
	4.3	7.8×10^{-6}	1800	0.873	1800	0.873
	5.1	4.0×10^{-6}	1800	0.871	1800	0.871
	5.9	2.4×10^{-6}	1800	0.872	1800	0.872

Test terminated because of insufficient cooling.

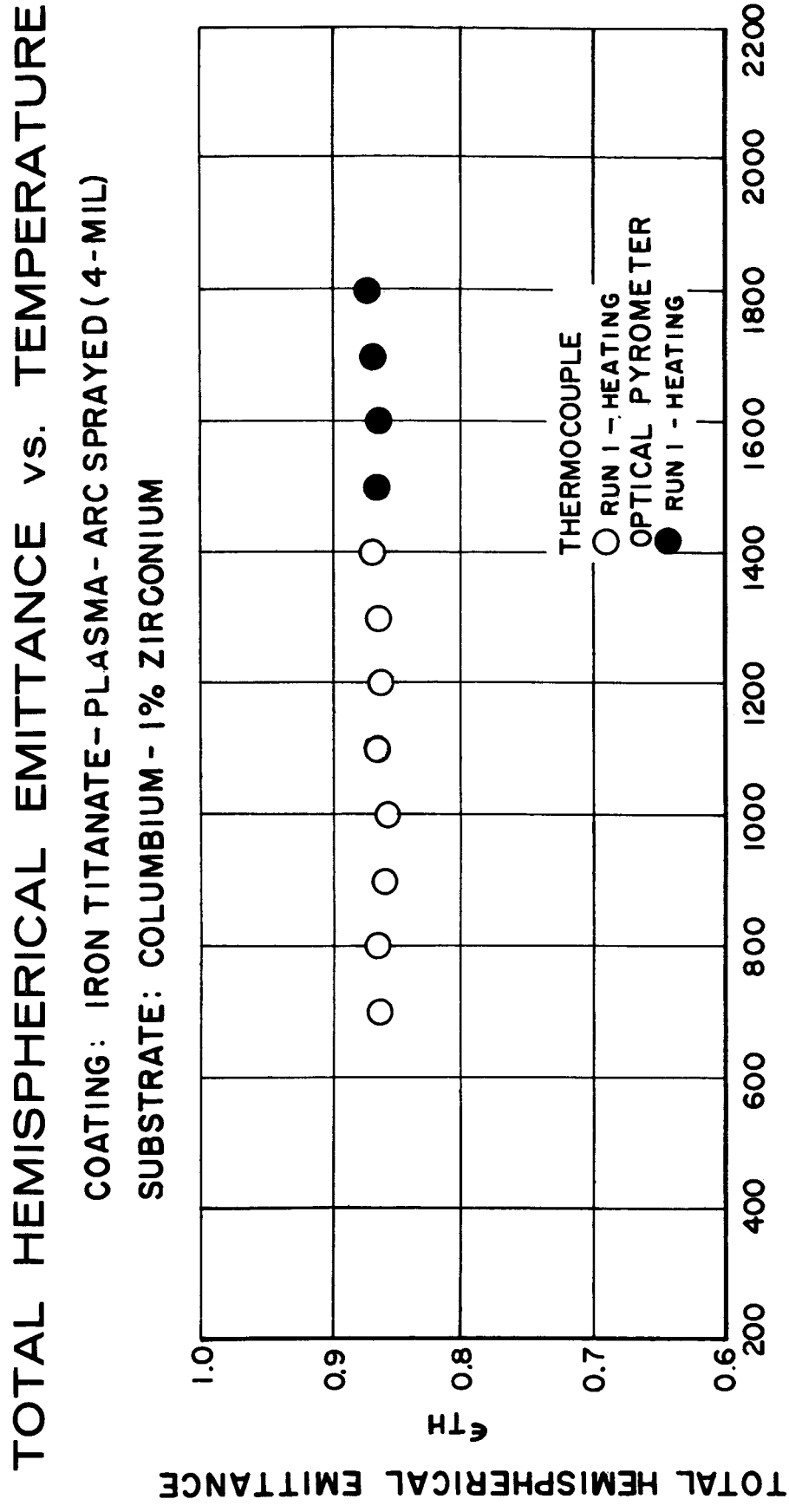

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE
COATING: IRON TITANATE-PLASMA-ARC SPRAYED (4-MIL)
SUBSTRATE: COLUMBIUM - 1% ZIRCONIUM
THERMOCOUPLE
RUN 1 - HEATING
OPTICAL PYROMETER
RUN 1 - HEATING
TOTAL HEMISPHERICAL EMITTANCE
εTH
1.0
0.9
0.8
0.7
0.6
200
400
600
800
1000
1200
1400
1600
1800
2000
2200
TEMPERATURE - °F

Figure 10

II. COATING ENDURANCE TESTS IN SUPPORT OF NASA SPACE POWER SYSTEMS

Work was continued during this reporting period in support of the SNAP-8 and Sunflower I space power systems. The endurance tests of each of the four finned-tube radiator segments scheduled to run ten thousand hours have been concluded. The specimens are at ambient temperature and will remain in vacuum pending post-test analysis.

A. Endurance Test No. 1, SNAP-8 Test Section

A mixture of nickel-chrome spinel ($NiO \cdot Cr_2O_3$) and silicon dioxide was aluminum-phosphate bonded to a SNAP-8 test section. As may be seen in Figure 11, the appearance of the coating has not changed since the inadvertent overheating at 2700 hours (see Technical Report PWA-2206). Testing was terminated after the specimen had accumulated 15,000 hours of exposure to a fin root temperature of 700°F at a pressure in the 10^{-8} mm Hg range. Cooling the specimen to the ambient temperature did not change its appearance (Figure 12).

B. Endurance Test No. 2, SNAP-8 Test Section
Endurance Test No. 3, Sunflower I Test Section

"Titania base" powder, obtained from the Plasmadyne Corporation, was plasma-arc sprayed onto a SNAP-8 and a Sunflower I radiator segment at Pratt & Whitney Aircraft using Plasmadyne powder spray equipment. The powder is titanium dioxide with small amounts of other oxides present.

The coating on the SNAP-8 test specimen continued to flake off the tube and more small spots of coating failure appeared during this report period. After approximately 12,287 hours the vacion pump stalled and shut off the power to the specimen. The appearance of the specimen during the period that the power was off is shown in Figure 13. Figure 14 shows the specimen after it was returned to endurance conditions. Comparison of Figures 13 and 14 shows that thermal cycling resulted in additional coating loss. Figures 15 and 16, respectively, show the specimen before and after it was cooled to the ambient temperature. When testing was concluded the specimen had accumulated a total of 14,037 hours of exposure to a fin root temperature of 650°F and a pressure in the 10^{-8} mm Hg range.

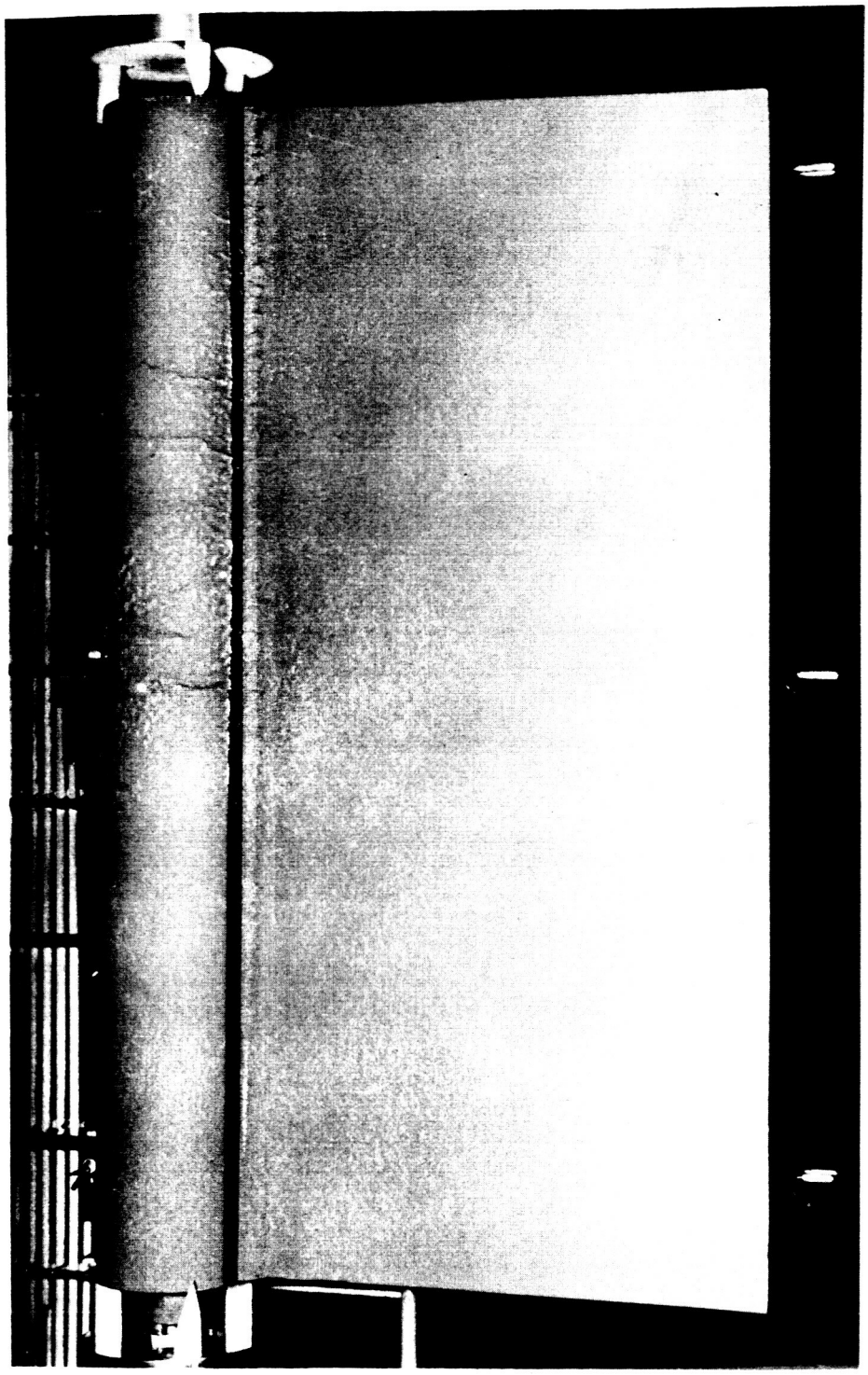
Power to the Sunflower I segment was shut off by the electromagnetic relay as a result of a line voltage fluctuation after 12,691 hours of endurance testing. The specimen was maintained in vacuum and examined before being returned to the endurance temperature. No change in the appearance of the coating resulted from the thermal cycling. After 13,755 hours the relay again shut off power to the specimen. Since endurance testing of the other specimens was to be concluded within the next two weeks it was decided not to continue testing the Sunflower I radiator segment. Figure 17 shows the appearance of the specimen at the ambient temperature after 13,755 hours of exposure to a fin root temperature of 650°F and a pressure in the 10^{-8} mm Hg range. No change in the appearance of the coating occurred as a result of testing.

C. Endurance Test No. 4, SNAP-8 Test Section

A mixture of silicon carbide and silicon dioxide was aluminum-phosphate bonded to this test section. Endurance testing was terminated after 12,781 hours of exposure to a fin root temperature of 700°F and a pressure in the 10^{-8} mm Hg range. As may be seen in Figures 18 and 19, no change in the appearance of the coating resulted from endurance testing or from cooling to the ambient temperature.

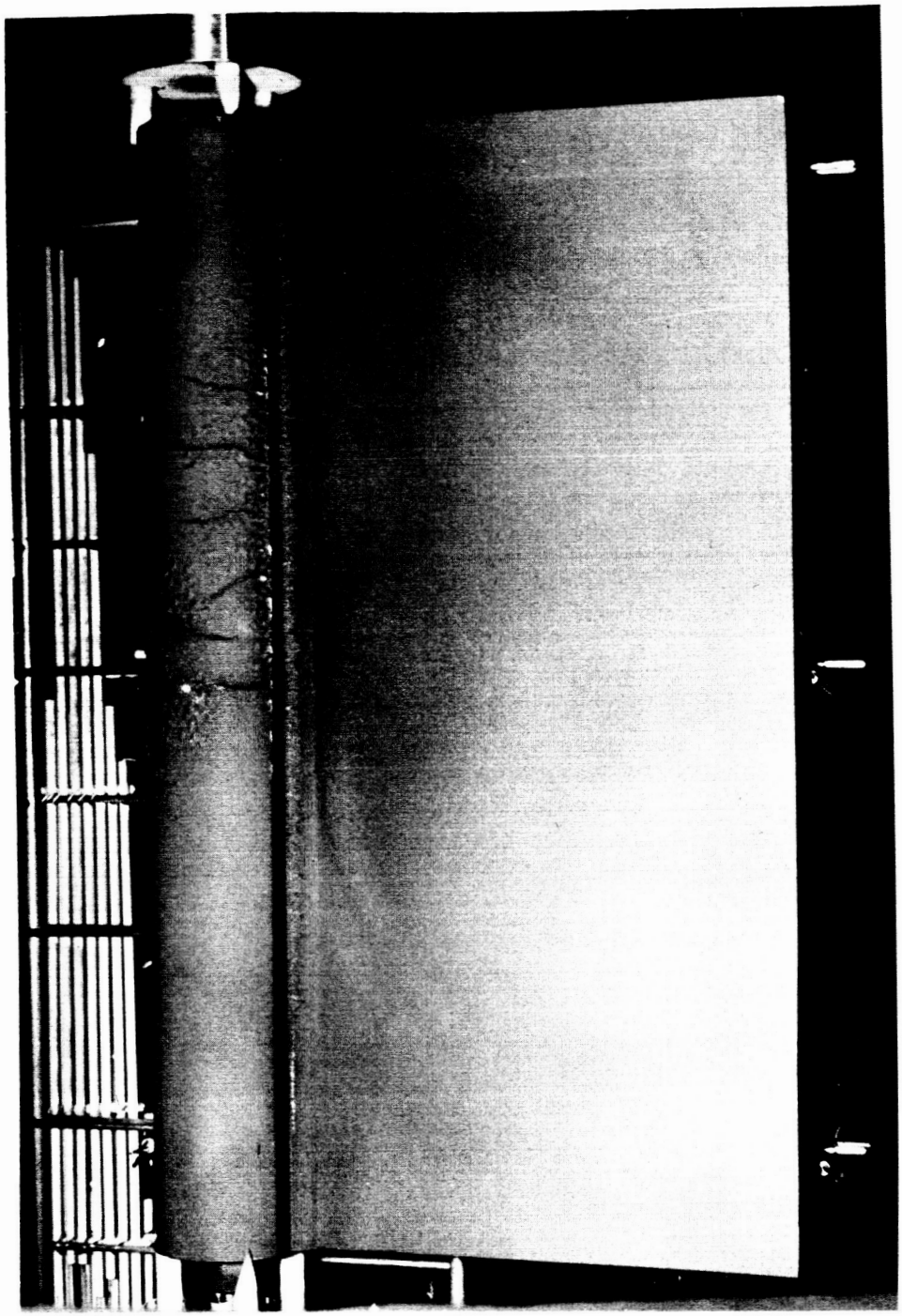
D. Scheduled Post-Test Analysis

Each of the long-term endurance specimens and the associated test equipment will be subjected to a thorough post-test analysis. Analysis of the test equipment will include a calibration check of the thermocouples and vac-ion pump and determination of the vacuum system leak rate. Analyses of the specimen will be concentrated at locations on the tube, tube-fin junction, fin midpoint, and near the fin tip although tests will be conducted at additional locations when warranted. Photomicrographs will be taken at each of these locations. The coatings will be tested for adhesion, flexibility, chemical composition, and structure. The substrate will be subjected to X-ray fluorescent inspection. These analyses will conclude the work in support of the SNAP-8 and Sunflower I space power systems.



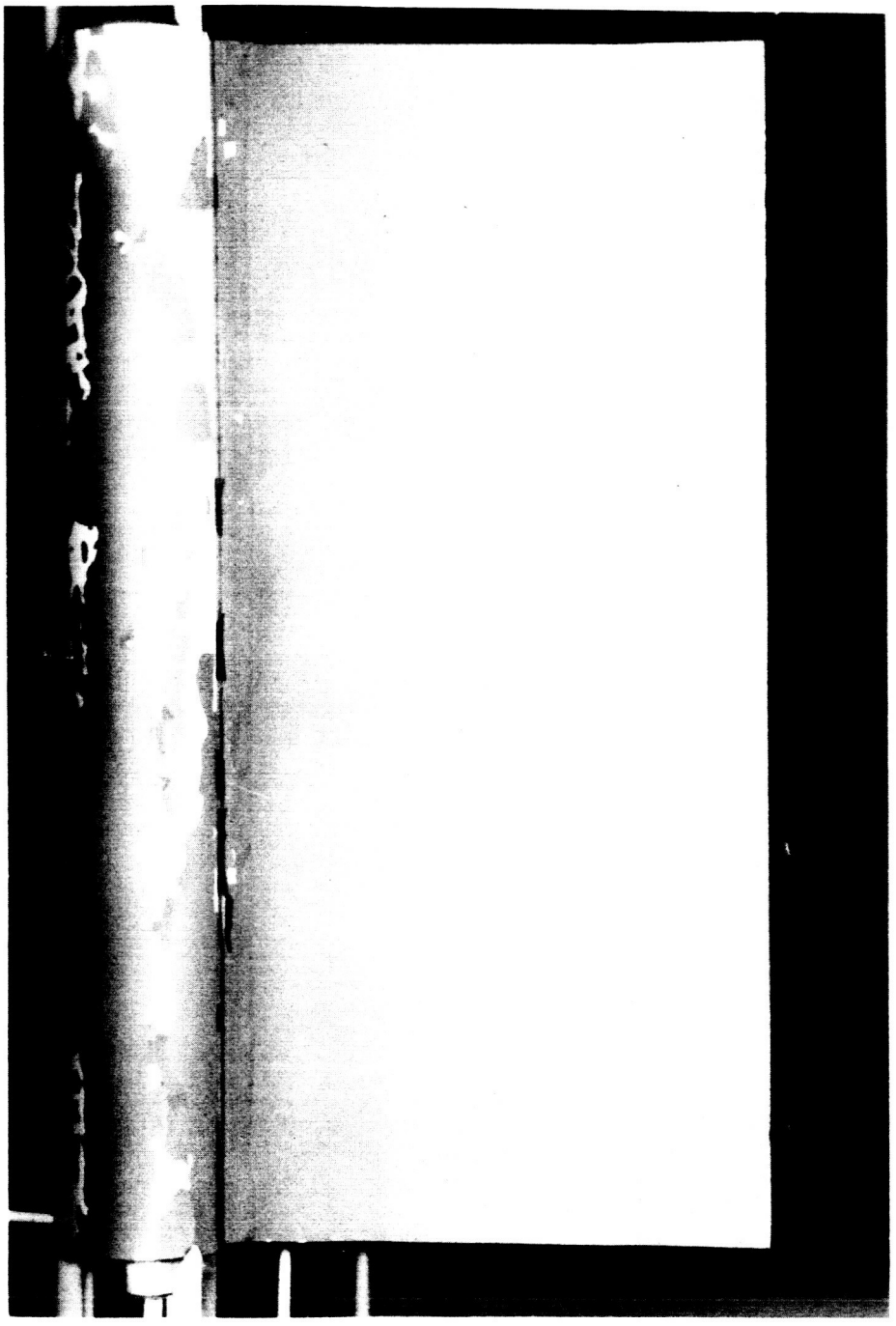
NICKEL-CHROME SPINEL COATED SNAP-8 FIN SEGMENT AT ENDURANCE CONDITIONS AFTER 14979 HOURS OF ENDURANCE TESTING.

Figure 11



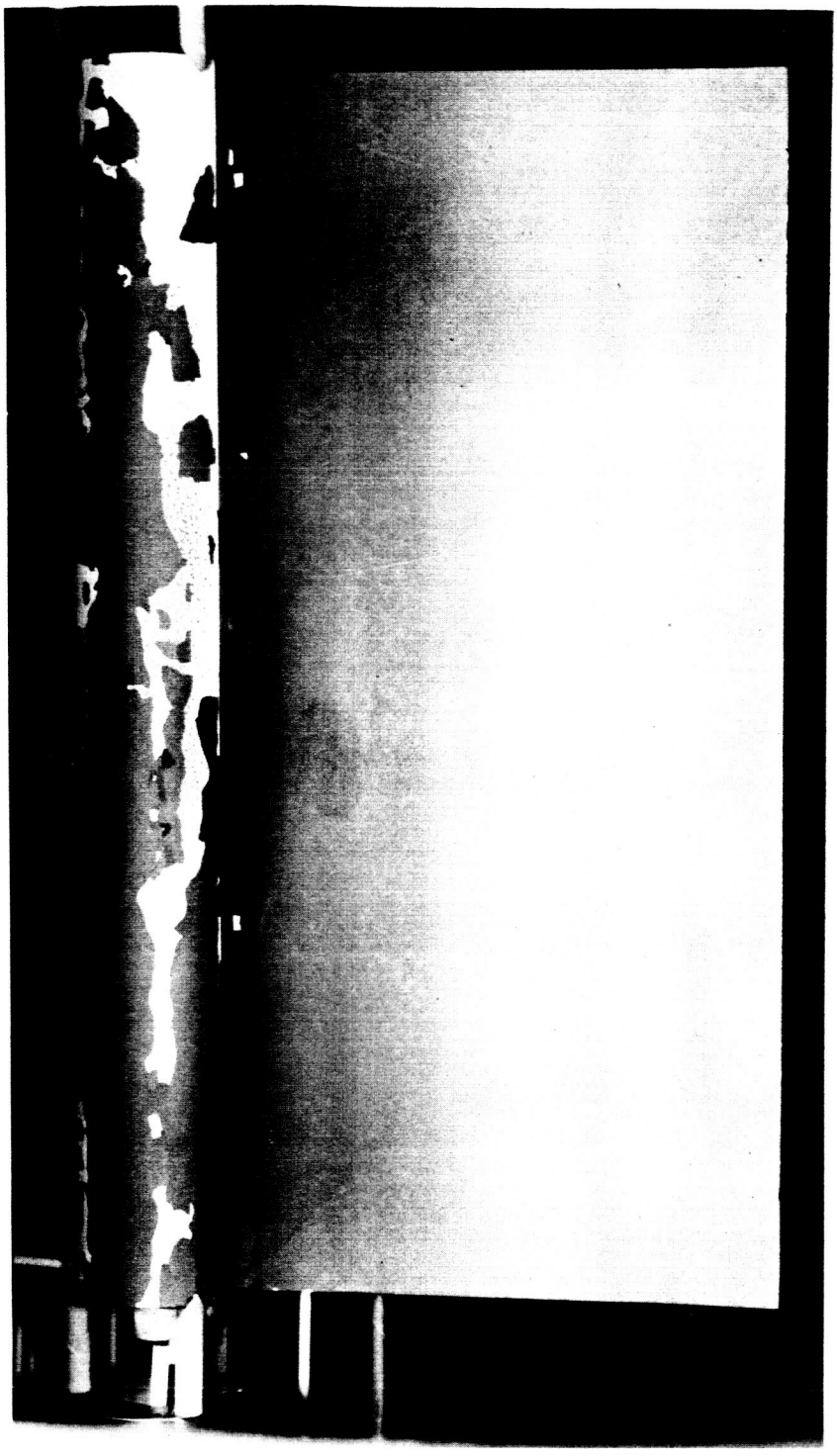
NICKEL-CHROME SPINEL COATED SNAP-8 FIN SEGMENT AFTER COMPLETION OF ENDURANCE TEST. TOTAL TIME AT ENDURANCE CONDITIONS WAS 15,000 HOURS.

Figure 12



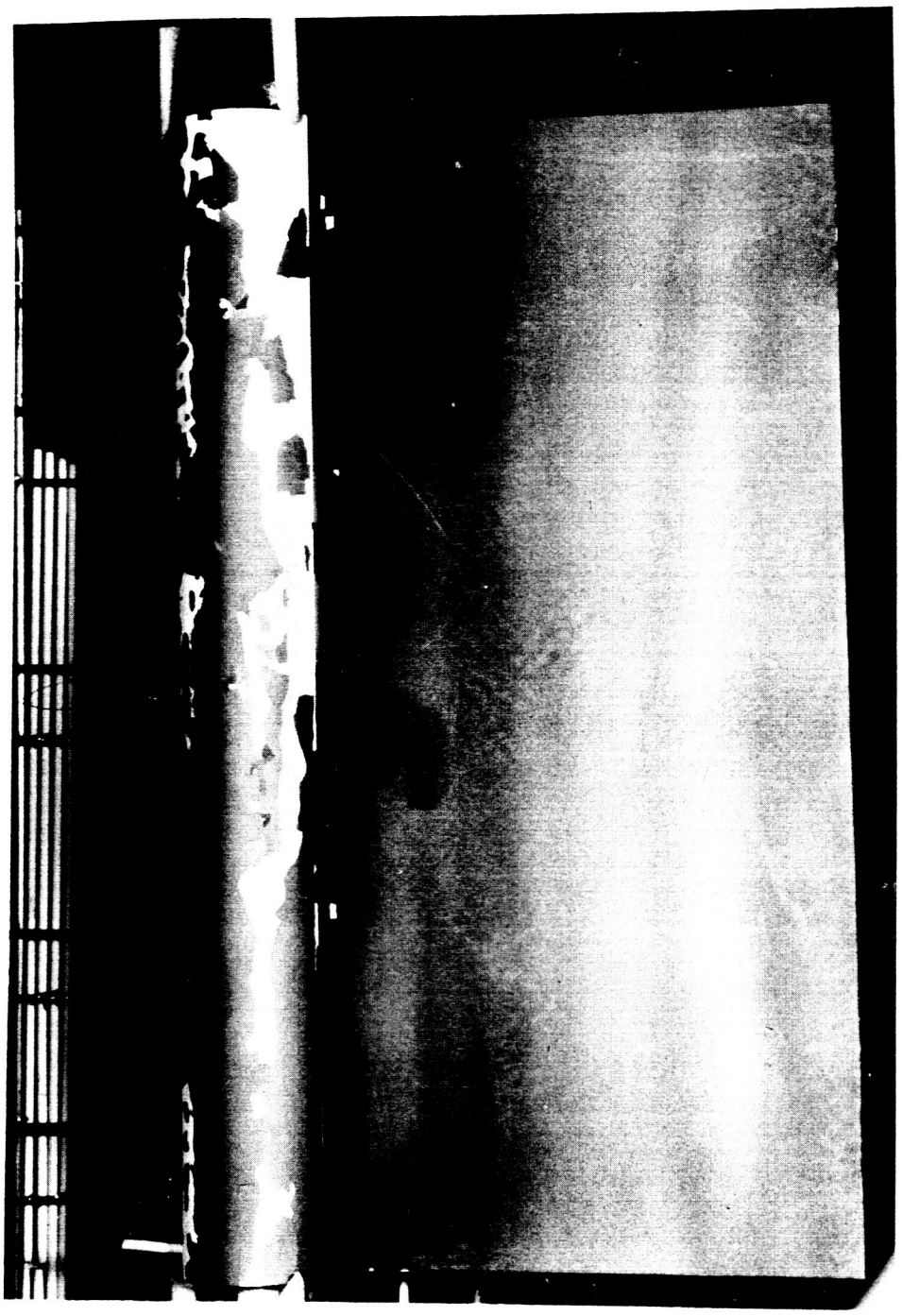
"TITANIA BASE" COATED SNAP-8 FIN SEGMENT WITH POWER SHUT OFF AFTER 12,287 HOURS OF ENDURANCE TESTING.

Figure 13



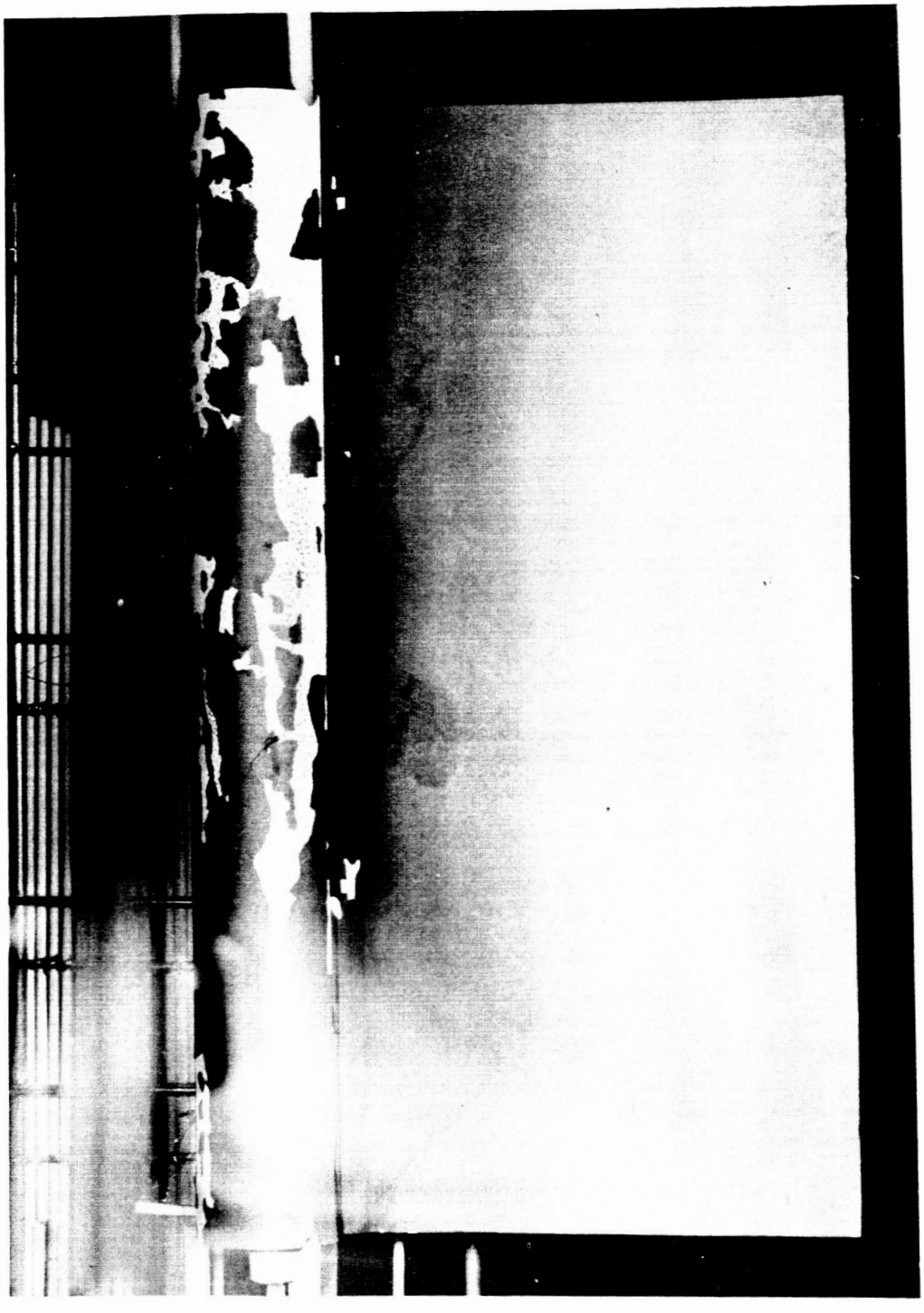
"TITANIA BASE" COATED SNAP-8 FIN SEGMENT AT ENDURANCE CONDITIONS AFTER POWER WAS SHUT OFF AT 12,287 HOURS.

Figure 14



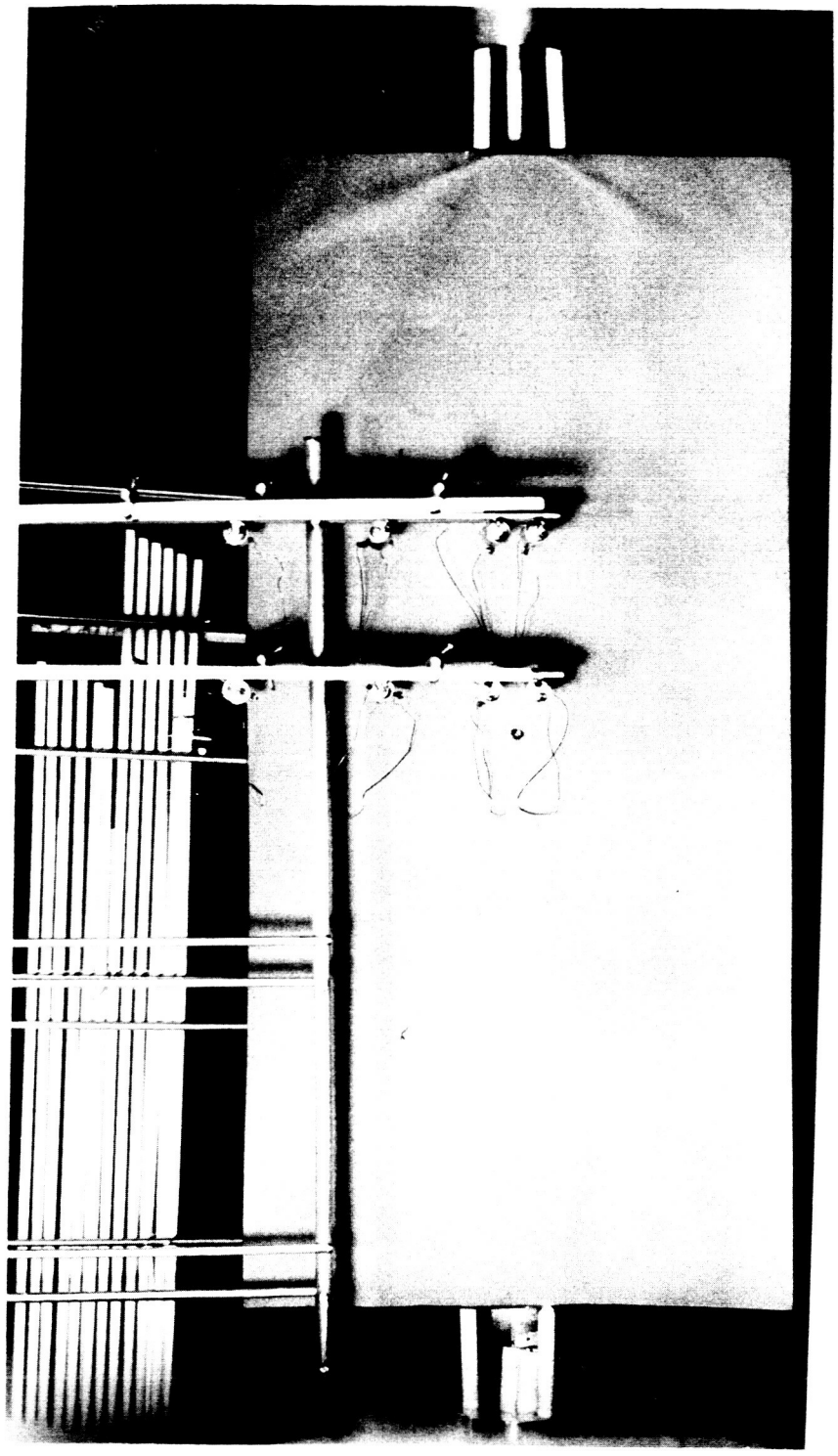
"TITANIA BASE" COATED SNAP-8 FIN SEGMENT AT ENDURANCE CONDITIONS AFTER 14017 HOURS OF ENDURANCE TESTING.

Figure 15



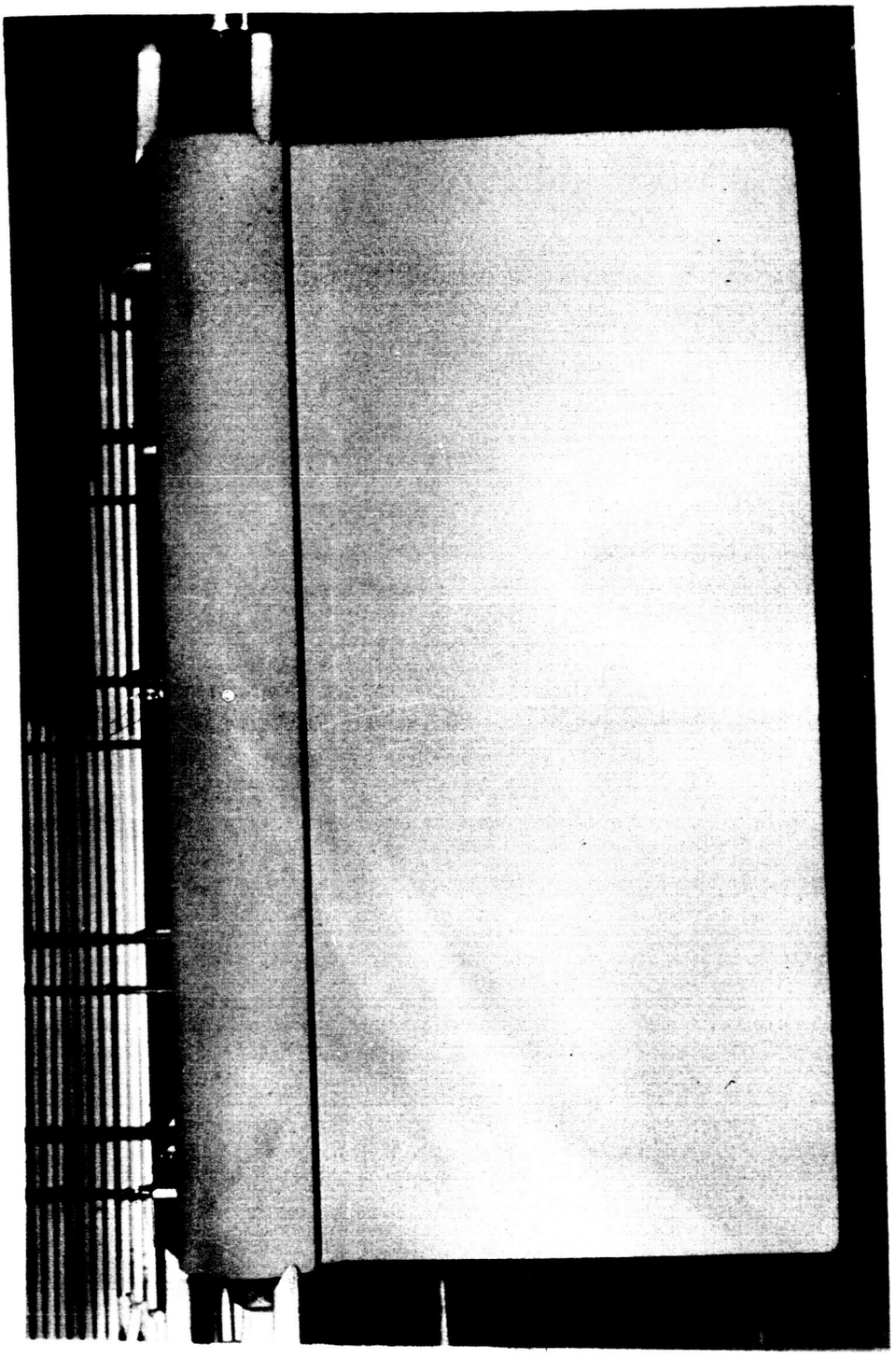
"TITANIA BASE" COATED SNAP-8 FIN SEGMENT AFTER COMPLETION OF ENDURANCE TEST. TOTAL TIME AT ENDURANCE CONDITIONS WAS 14037 HOURS.

Figure 16



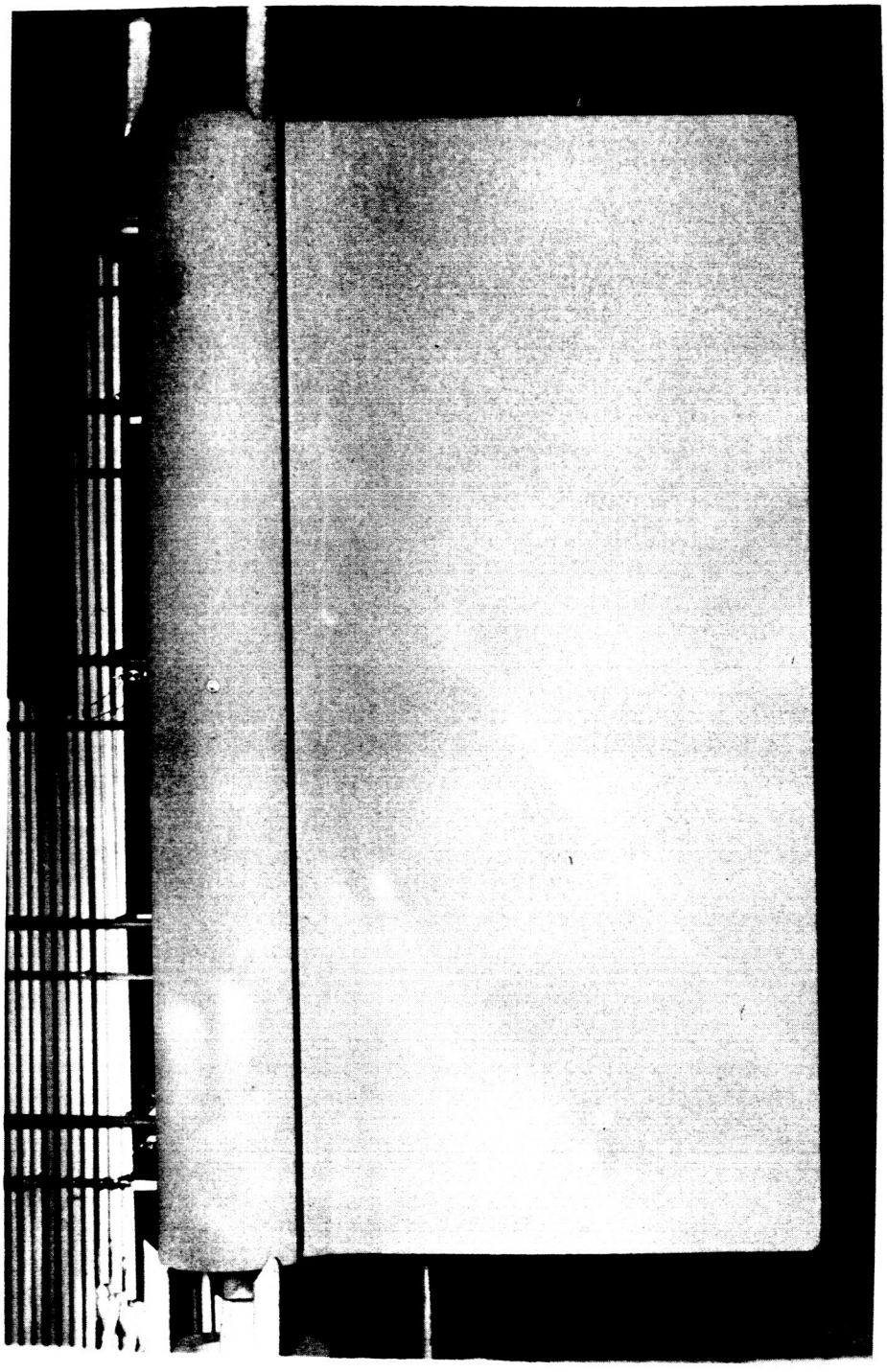
"TITANIA BASE" COATED SUNFLOWER- 1 FIN SEGMENT AFTER COMPLETION OF ENDURANCE TEST. TOTAL TIME AT ENDURANCE CONDITIONS WAS 13755 HOURS.

Figure 17



SILICON CARBIDE COATED SNAP-8 FIN SEGMENT AT ENDURANCE CONDITIONS AFTER 12762 HOURS OF ENDURANCE TESTING.

Figure 18



SILICON CARBIDE COATED SNAP-8 FIN SEGMENT AFTER COMPLETION OF ENDURANCE TEST. TOTAL TIME AT ENDURANCE CONDITIONS WAS 12781 HOURS.

Figure 19

III. INVESTIGATION OF ALKAPHOS-BONDED COATING PROCEDURES

Difficulty has been encountered in maintaining adequate bonding with aluminum-phosphate bonded coatings at temperatures above 1400°F. Although most high-temperature emittance coatings may be satisfactorily applied by thermal spraying, silicon carbide, which has recently been found to have an emittance higher than 0.90, decomposes when applied by thermal spraying. Conventional coating techniques other than aluminum phosphate bonding have not been found suitable for high temperature space radiator coatings. Since a stable aluminum phosphate compound, anhydrous aluminum metaphosphate ($Al_2O_3 \cdot 3P_2O_5$) is formed at 930°F[1] with the loss of the last of the chemically combined water, it would be expected that, in the absence of thermal shocking and below 2000°F, bonds would either fail below 930°F or not at all. Since bonding failures have occurred above 930°F, an investigation of substrate surface preparation and curing procedures is being conducted.

Since the source of aluminum-phosphate solution used for bonding has not been found to influence emittance data, the investigation has been confined to one commercially available solution, namely, Alkaphos C, a product of Monsanto Chemical Company. This product is particularly stable which enables a single batch of material to be used throughout the investigations and therefore precludes the possibility of variations in mixing practice influencing the test results. AISI-310 stainless steel strips and columbium-1 per cent zirconium tubes have been used for substrates.

To determine the effects of surface roughness, four columbium-1 per cent zirconium tubes were prepared with varying degrees of roughness. The processes used were chemical cleaning, vapor blasting, grit blasting with 90 mesh alumina, and grit blasting with 28 mesh steel. Each tube was cleaned with trichloroethylene, flushed with water, and rinsed with acetone. The coating and curing procedure used is outlined in Table IX, specimens numbers 1 through 4. Although some crumbling occurred on all of the samples, the coating applied to the tube which was grit blasted with 90 mesh alumina showed the least tendency to separate from its substrate.

[1]Eubanks, A. G. and Moore, D. G., "Investigation of Aluminum Phosphate Coatings for Thermal Insulation of Airframes," NASA TN D-106, National Aeronautics & Space Administration, Washington, D.C., November, 1959.

Various curing cycles were investigated for Alkaphos-bonded silicon-carbide coatings on AISI-310 stainless steel strips and on columbium-1 per cent zirconium tubes. The substrates were prepared by either vapor or grit blasting and were degreased and rinsed with acetone immediately before being coated. Coatings of various compositions (see Table IX) were applied by spraying and were dried and cured for various times and at various temperatures. All of the specimens which were heated above 400°F were furnace-cooled to 400°F to prevent thermal shocking. Results of these tests appear in Table IX, specimens numbers 5 through 14. Coatings which were cured at temperatures above 400°F remained intact until after the specimens were removed from the furnace, but after a few hours the coatings crumbled. Since this problem had not been encountered previously when a more acidic aluminum-phosphate solution made by Pratt & Whitney Aircraft had been used, it was thought that increasing the acidity of the Alkaphos might reduce the amount of crumbling. A specimen was prepared using a slurry to which phosphoric acid had been added but excessive spalling of the coating resulted (specimen number 15 in Table IX). To gain further insight into the problem, a coating with $SrO \cdot TiO_2$ filler was applied and cured at a temperature above 400°F. No crumbling occurred (Table IX, specimen number 16).

As may be seen in Table IX, all of the silicon carbide coatings bubbled during curing. In an attempt to determine the cause of the bubbling, coatings of Alkaphos C without filler material were applied to a stainless steel and to a columbium-1 per cent zirconium substrate. Curing included heating to 950°F. No bubbling occurred, but the coatings were still tacky at completion of the curing cycle indicating incomplete curing (see specimens 17 and 18 in Table IX). The lack of bubbling, however indicates that the Alkaphos C does not, in itself, cause the bubbling. It was noted that the higher-purity green silicon carbide was not so prone to bubbling as was the black silicon carbide. On this basis it is considered possible that the bubbling is caused by impurities which react with the aluminum phosphate solution.

To further evaluate the cause of bubbling, a coating containing $CaO \cdot TiO_2$ filler material was applied to a columbium-1 per cent zirconium tube. This specimen (specimen number 19 in Table IX) was cured by the process which had been most successful with silicon carbide coatings. No bubbling occurred.

On the basis of work completed to date, and consultation with the Monsanto Chemical Company, it appears that silicon carbide is a particularly difficult material to bond by aluminum phosphate solutions. The best coatings produced to date were air-dried for 20 hours, and oven cured at 200°F for 2 hours, 250°F for 2 hours, 300°F for 2 hours, and 400°F for 2 hours. The best SiC and $CaO \cdot TiO_2$ coatings produced will be tested in the total hemispherical emittance rig to determine their stability at temperatures in excess of 1400°F.

TABLE IX

Results of Investigation of Alkaphos C-Bonded Coating Procedures

Specimum Number	Coating	Surface Preparation	Substrate	Slurry Composition	Air Drying Time (Hours)	Curing Cycle *	Results	Remarks
1	Black SiC	Chemically Cleaned	Cb-1Zr	100 gms SiC 100 ml Alkaphos	66	180F(1) + 220F(1) + 300F (2)	No change noted after 180F+ 220F cycle. Steel grit blasted tube coating was very rough. After 300F cycle all tubes showed evidence of bubbling with Al_2O_3 grit blasted tube showing the least.	Specimens were furnace cooled from 700F in about four hours. Coatings exhibited crust-like surface (bubbling). After standing in cabinet about 2 hours coatings spalled off of all 4 tubes with the the Al_2O_3 grit blasted
2	Black SiC	Vapor Blasted	Cb-1Zr					
3	Black SiC	Grit Blasted-90 Grit Al_2O_3	Cb-1Zr					
4	Black SiC	Grit Blaster-28 Grit Steel	Cb-1Zr					
						375F(1 1/2) + 700F(3)	More evidence of bubbling after two latter cycles.	
5	Black SiC	Vapor Blasted	Stainless Steel	100 gms SiC 100 ml Alkaphos	2	400F(2) + 900F(2)	No bonding. Light, medium and heavy coatings were powdery after curing.	Medium and heavy coatings showed evidence of bubbling.
6	Black SiC	Vapor Blasted	Stainless Steel	100 gms SiC 100 ml Alkaphos	20	400F(2) + 900F(2)	No bonding. Light, medium and heavy coatings were powdery after curing.	Medium and heavy coatings showed evidence of bubbling.
7	Black SiC	Vapor Blasted	Stainless Steel	100 gms SiC 100 ml Alkaphos	48	300-350F(64)	No bond between coating and substrate after curing. Coating was uniform and could not be rubbed off of substrate after air dry.	Coating came off in one complete sheet. Good bond between particles. Evidence of bubbling.
8	Black SiC	Vapor Blasted	Stainless Steel	100 gms SiC 100 ml Alkaphos	100	180-250F(10) + 500F(2) + 950F(2)	No bonding between coating and substrate.	Coating came off in complete sheet. Good bond between particles. Evidence of bubbling.
9	Black SiC	Vapor Blasted	Stainless Steel	100 gms SiC 100 ml Alkaphos	336	200F(2) + 260F(15) + 300F(2) + 400F(2) + 700F(3)	Coating showed no bubbling during first 4 cycles.	Coating crumbled after cooling and setting in air for 24 hours.
10	Black SiC	Grit Blasted-90 Grit Al_2O_3	Stainless Steel	100 gms SiC 100 ml Alkaphos	24	250F(10)+500F(2)+ 950F(2)	No bond between coating and substrate.	

* Numbers in parenthese indicate hours at temperature

TABLE IX (Cont'd.)

Specimen Number	Coating	Surface Preparation	Substrate	Slurry Composition	Air Drying Time (Hours)	Curing Cycle*	Results	Remarks
11	Black SiC	Grit Blasted-90 Grit Al_2O_3	Cb-1Zr	100 gms SiC 100 ml Alkaphos	24	250F(10) + 500F(2) + 950F(2)	Coating adhered quite well	Substrate oxidized at 900F. Evidence of bubbling.
12	Black SiC	Grit Blasted-90 Grit Al_2O_3	Cb-1Zr	100 gms SiC 100 ml Alkaphos	68	200F(2) + 250F(15)+ 300F(2) + 400F(2) + cooled + 700F(2)	Coating bubbled slightly after 300F cycle. Coating was hard and intact after 700F before furnace cool.	Coating was hard and intact. Could not be scraped from tube except in bubbled areas after 400F cycle.
13	Green SiC	Grit Blasted - 90 Grit Al_2O_3	Cb-1Zr	100 gms SiC 100 ml Alkaphos	20	200F(2) + 250F(2) + 300F(2) + 400F(2) + cooled + 700F(2)	Bubbled slightly after 400F cure. Excessive spalling and breakdown of bond after 700F.	
14	Green SiC	Grit Blasted-90 Grit Al_2O_3	Cb-1Zr	100 gms SiC 100 ml Alkaphos	20	200F(2) + 250F(2) + 300F(2) + 400F(2)	Coating showed slight bubbling after 400F cycle but otherwise exhibited good bonding.	
15	Green SiC	Grit Blasted - 90 Grit Al_2O_3	Cb-1Zr	100 gms SiC 100 ml Alkaphos 10 ml H_3PO_4	20	200F(2) + 250F(2) + 300F(2) + 400F(2) + cooled + 700F(2)	Did not cure during cycles to 400F. Excessive spalling after 700F.	
16	$SrTiO_2$	Grit Blasted - 90 Grit Al_2O_3	Cb-1Zr	100 gms $SrTiO_3$ 125 ml Alkaphos	20	200F(2) + 250F(2) + 300F(2) + 400F(2) + cooled + 700F(2)	Very strong bond of coating to substrate. No evidence of bubbling.	Coating was white after 400F cycle but had a brownish tinge after 700F cycle. Coating seemed to be more brittle after 700F cure.
17	Alkaphos C	Grit Blasted-90 Grit Al_2O_3	Stainless Steel	No filler	90	250F(10) + 500F(2) + 950F(2)	Coating was white and flaky and could be scraped off. Little evidence of bubbling.	Coating was tacky after drying. Showed areas of incomplete wetting.
18	Alkaphos C	Grit Blasted-90 Grit Al_2O_3	Cb-1Zr	No filler	90	250F(10) + 500F(2) + 950F(2)	Color varied from transparent in thin areas to white in thick areas. Coating could be scraped off but had little bubbling.	Coating was tacky after drying. Showed areas of incomplete wetting.
19	$CaTiO_3$	Grit Blasted-90 Grit Al_2O_3	Cb-1Zr	100 gms $CaTiO_3$ 150 ml Alkaphos	20	200F(2) + 250F(2) + 300F(2) + 400F(2)	Good bond between coating and substrate. Coating turned reddish color on exposed top side. No bubbling.	Coating difficult to apply because of settling of particles in solution.

* Numbers in parentheses indicate hours at temperature

IV. INVESTIGATION OF TOTAL HEMISPHERICAL EMITTANCE RIG DISCREPANCIES

As reported previously in Pratt & Whitney Aircraft Technical Report PWA-2163, difficulty had been encountered in reconditioning the total hemispherical emittance rig after a coating of manganese oxide volatilized and coated the instrumentation flange and other parts in the chamber with a metallic coating. A complete cleaning of the interior of the chamber and replacement of the affected electrical components failed to re-establish the previous performance. Although measurements of low emittances agreed with those previously obtained to within less than 1 per cent, measurements of high emittances consistently resulted in values which were higher than those obtained previously or obtained in the other rigs. Since total hemispherical emittance measurements depend on measurements of specimen power input and specimen surface temperature, the components involved with these measurements were carefully analyzed. Power is measured in this rig with a Fluke voltmeter in conjunction with several current shunts. The voltmeter used was checked and found to be accurate to within 0.2 per cent and the current shunts were checked and found to deviate from their nominal values by not more than 0.08 per cent.

An analysis of temperature measuring equipment, however, revealed that temperature values obtained with chromel-alumel thermocouples were, on the average, 6°F lower than those obtained with the optical pyrometer. Further, values obtained with platinum-platinum 10 per cent rhodium thermocouples (which are used extensively in the total hemispherical emittance rig) were consistently 20°F lower than those obtained with chromel alumel thermocouples. This 20°F discrepancy in temperature measurement is consistent with the discrepancies in emittance which are proportional to the emittance being measured. Initial investigation of the thermocouples evaluated the effect of the substrate material on thermocouple operation. Both platinum-platinum 10 per cent rhodium and chromel-alumel thermocouples were attached to uncoated tubes of columbium-1 per cent zirconium, AISI-310 stainless steel, and tantalum. It was found that the temperature measurement discrepancy was the same with all substrates and it was therefore concluded that the substrate material was not the cause of the discrepancy.

Analysis was then directed to the thermocouple wire itself, and it was discovered that one roll had not been properly annealed. This roll had been started at the time of the volatilization of the manganese oxide in

the total hemispherical emittance rig. To confirm that the improper preparation of the wire was the cause of the discrepancy, temperature measurements were made using 3-mil diameter chromel-alumel thermocouples and both 1-mil and 3-mil diameter platinum-platinum 10 per cent rhodium thermocouples which were properly annealed. Agreement between the 3-mil diameter platinum-platinum 10 per cent rhodium and the chromel-alumel thermocouples was within a few degrees in the range of 200 to 1800°F (see Table X). As shown in Table XI, agreement was also good between the 1- and 3-mil diameter platinum-platinum 10 per cent rhodium thermocouples, thus confirming the theoretical analysis of thermocouple lead heat conduction losses presented in Appendix L of Pratt & Whitney Aircraft Technical Report PWA-2206. In conjunction with the thermocouple investigations, emittance data was obtained for AISI-310 stainless steel and tantalum and are reported in sections I-A and I-B of this report.

The results of this investigation have resolved the difficulties arising from the volatilization of manganese oxide in the total hemispherical emittance rig and the rig has been returned to service.

As mentioned previously, it was discovered after a large number of temperature readings had been taken that, on the average, the optical pyrometer indicates a temperature which is 6°F higher than that indicated by thermocouples. Since the optical pyrometer had been calibrated during this investigation against another optical pyrometer and against a ribbon filament lamp, both with calibrations traceable to the National Bureau of Standards, it was deemed necessary to investigate the quality of the black-body holes being used in greater detail than previously. Previous investigations have been discussed in Technical Report PWA-2206.

Three different hole sizes were investigated, all in uncoated columbium-1 per cent zirconium tubes with 0.010-inch thick walls. Temperatures were measured by thermocouples and by the optical pyrometer over the temperature range of 1500°F to 1900°F. Temperatures were limited to below 1900°F to preclude failure of the chromel-alumel thermocouples (see Appendix N of Technical Report PWA-2206). Tests were run with the interiors of the tubes uncoated and also with the interiors coated with acetylene black in xylol. The results are plotted as optical pyrometer value minus thermocouple value (ΔT) versus specimen temperature.

Figure 20 shows the results of measurements obtained using a single rectangular black-body hole measuring 0.076 by 0.035 inch. Blackening of the tube interior definitely improved the black-body quality as indicated by the increase in optical pyrometer temperature indications.

Figure 21 shows the results obtained using a single 0.040-inch diameter black-body hole. Blackening the interior of this tube did not significantly affect the optical pyrometer indications and it is concluded therefore that the black-body hole quality cannot be improved by raising the emittance of the cavity walls. Comparison of Figures 20 and 21 reveals that between 1500 and 1800°F the quality of the 0.040-inch diameter hole is somewhat better than that of the blackened rectangular hole but that between 1800°F and 1900°F the two are about equivalent.

The black-body hole configuration normally used for emittance specimens is two circular holes 0.0235 inch in diameter. The results obtained using this configuration are shown in Figure 22 and average values appear in Figure 23. Since the level of optical pyrometer readings was not increased by blackening the interior of the tube, and since the level obtained was about the same as that obtained using the 0.040-inch diameter hole, it is concluded that these black-body holes are of sufficient quality that no significant increase in the optical pyrometer indications can be obtained by further improvement in the black-body hole quality. As shown in Figure 23, the optical pyrometer values average about 6°F higher than the thermocouple values.

For one series of measurements the black-body holes were rotated off-axis in the wrong direction so that the holes did not present a maximum cross-section to the optical pyrometer. Although the reduced cross-section made temperature measuring more difficult, it did not result in different values and it may therefore be concluded that minor misalignment of the axis of the black-body holes does not affect the pyrometer indications. The chamber of the rig was then rotated 45° to move the specimen off the centerline between the rig windows. The results of temperature measurements with this configuration appear in Figure 24 and indicate that misalignment of the specimen within the chamber does not significantly affect optical pyrometer indications.

The final portion of this investigation was carried out in the short term endurance rig to confirm the effects of chamber geometry and to allow the use of a thermodot (an infrared pyrometer) as another means of temperature measurement. The results obtained from this series appear in Figure 25. These results are in agreement with the results obtained from the total emittance rig and confirm the conclusion that the rig geometry has little or no effect on the discrepancy between optical pyrometer and thermocouple temperature indications.

Although the cause of the 6°F discrepancy has not been found, this investigation has shown that the discrepancy is not a result of effects of the black-body cavity, specimen misalignment, or chamber geometry. Since 6°F introduces a maximum uncertainty of only 1.1 per cent into the values of total hemispherical emittance, emittance measurements are being continued pending resolution of the problem.

TABLE X

Temperature Values Obtained With 3-Mil Diameter Thermocouples on Uncoated Polished Tantalum

Platinum-Platinum 10% Rhodium		Chromel-Alumel
Improperly Annealed Condition Temperature Reading (°F)	Fully Annealed Condition Temperature Reading (°F)	As-Received Condition Temperature (°F)
196	200	199
393	400	400
592	601	600
790	800	800
990	1000	1000
1191	1199	1200
1393	1400	1400
1596	1600	1601
1800	1801	1794

TABLE XI

Temperature Values Obtained with Fully Annealed 1- and 3-Mil Diameter Platinum-Platinum 10 Per Cent Rhodium Thermocouples on Uncoated, Polished Tantalum

Temperature Reading with 1-Mil Diameter Wire (°F)	Temperature Reading with 3-Mil Diameter Wire (°F)
999	999
1195	1194
1399	1396
1604	1602
1809	1808
2006	2006
2207	2204

DIFFERENCE BETWEEN OPTICAL PYROMETER AND THERMOCOUPLE TEMPERATURE INDICATIONS vs. SPECIMEN TEMPERATURE USING ONE RECTANGULAR BLACK - BODY HOLE 0.035" x 0.076"

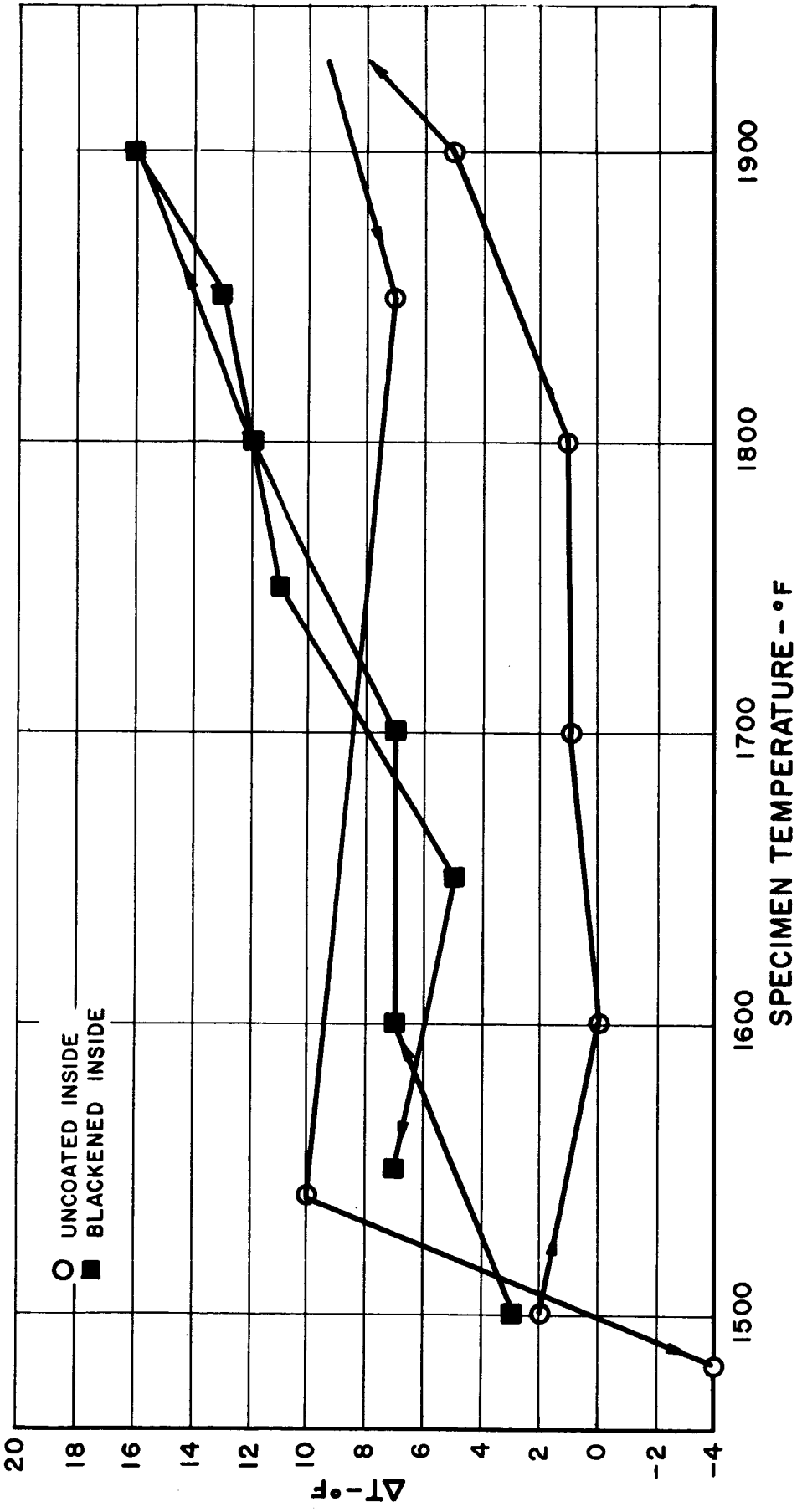


Figure 20
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DIFFERENCE BETWEEN OPTICAL PYROMETER AND THERMOCOUPLE TEMPERATURE INDICATIONS vs. SPECIMEN TEMPERATURE USING ONE CIRCULAR BLACK-BODY HOLE 0.040" IN DIAMETER

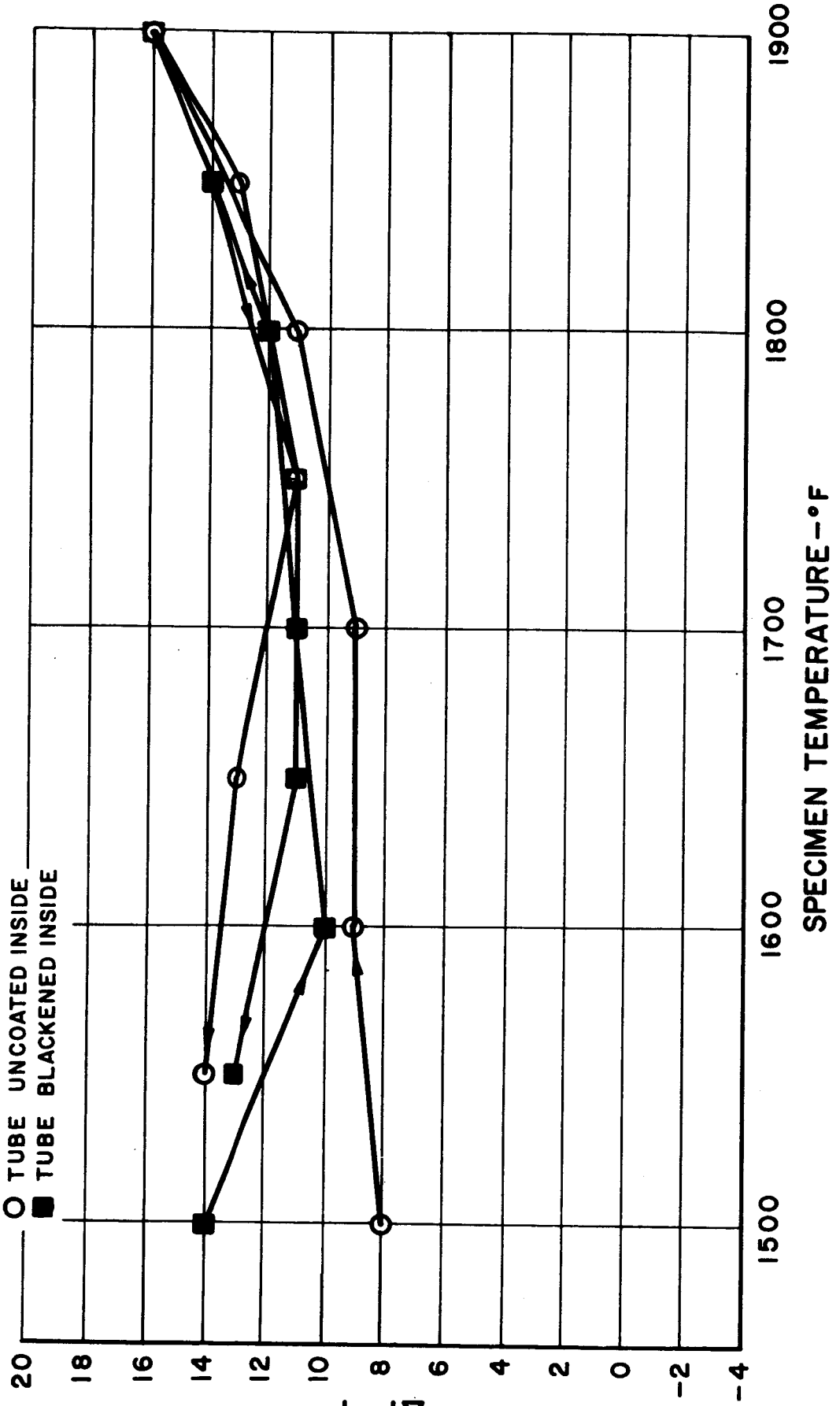

Figure 21
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DIFFERENCE BETWEEN OPTICAL PYROMETER AND THERMOCOUPLE TEMPERATURE INDICATIONS vs. SPECIMEN TEMPERATURE USING TWO CIRCULAR BLACK - BODY HOLES 0.023" IN DIAMETER

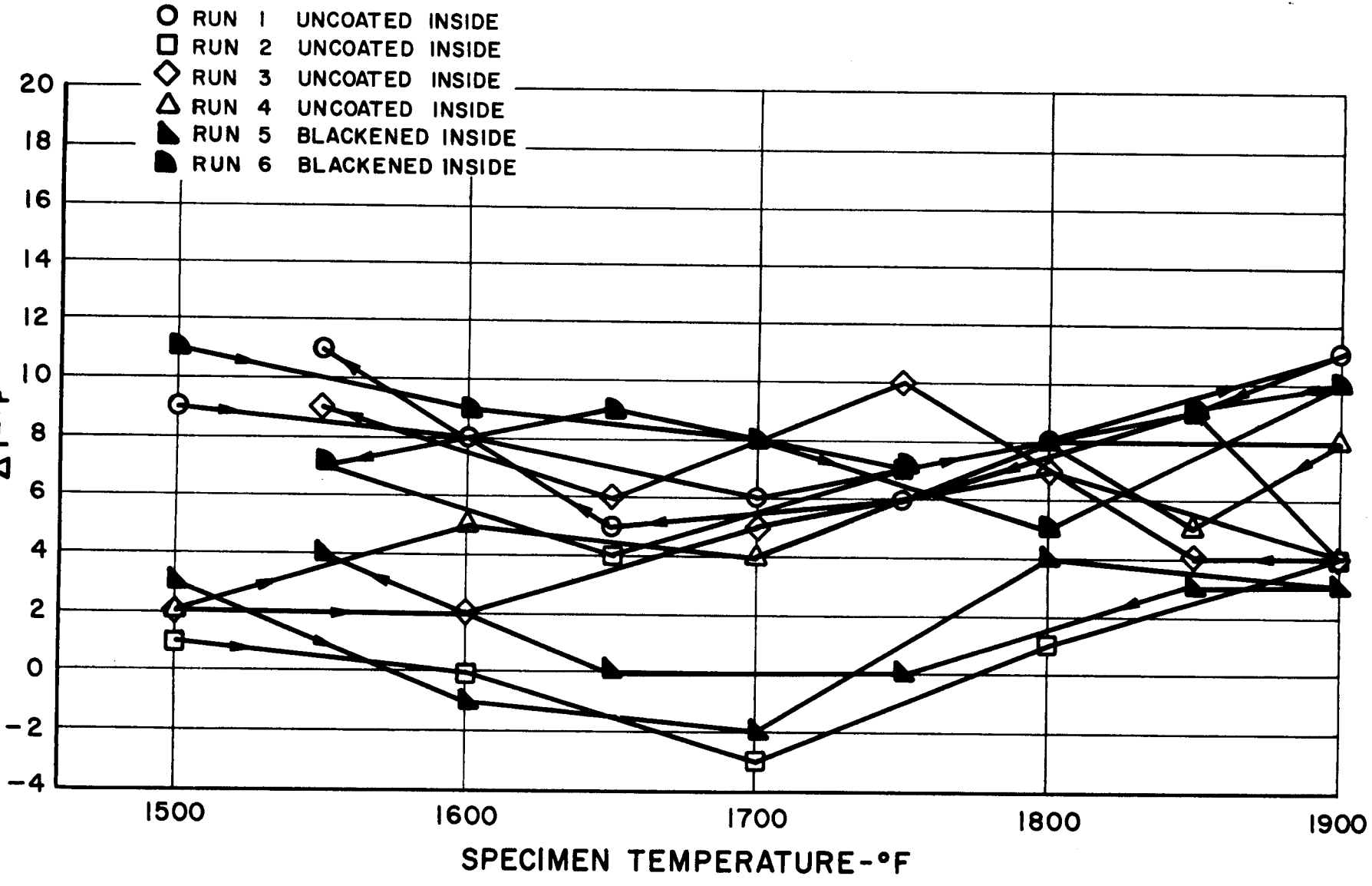

Figure 22
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AVERAGE VALUES OF THE DIFFERENCE BETWEEN OPTICAL PYROMETER AND THERMOCOUPLE TEMPERATURE INDICATIONS vs. SPECIMEN TEMPERATURE USING TWO CIRCULAR BLACK - BODY HOLES 0.023" IN DIAMETER

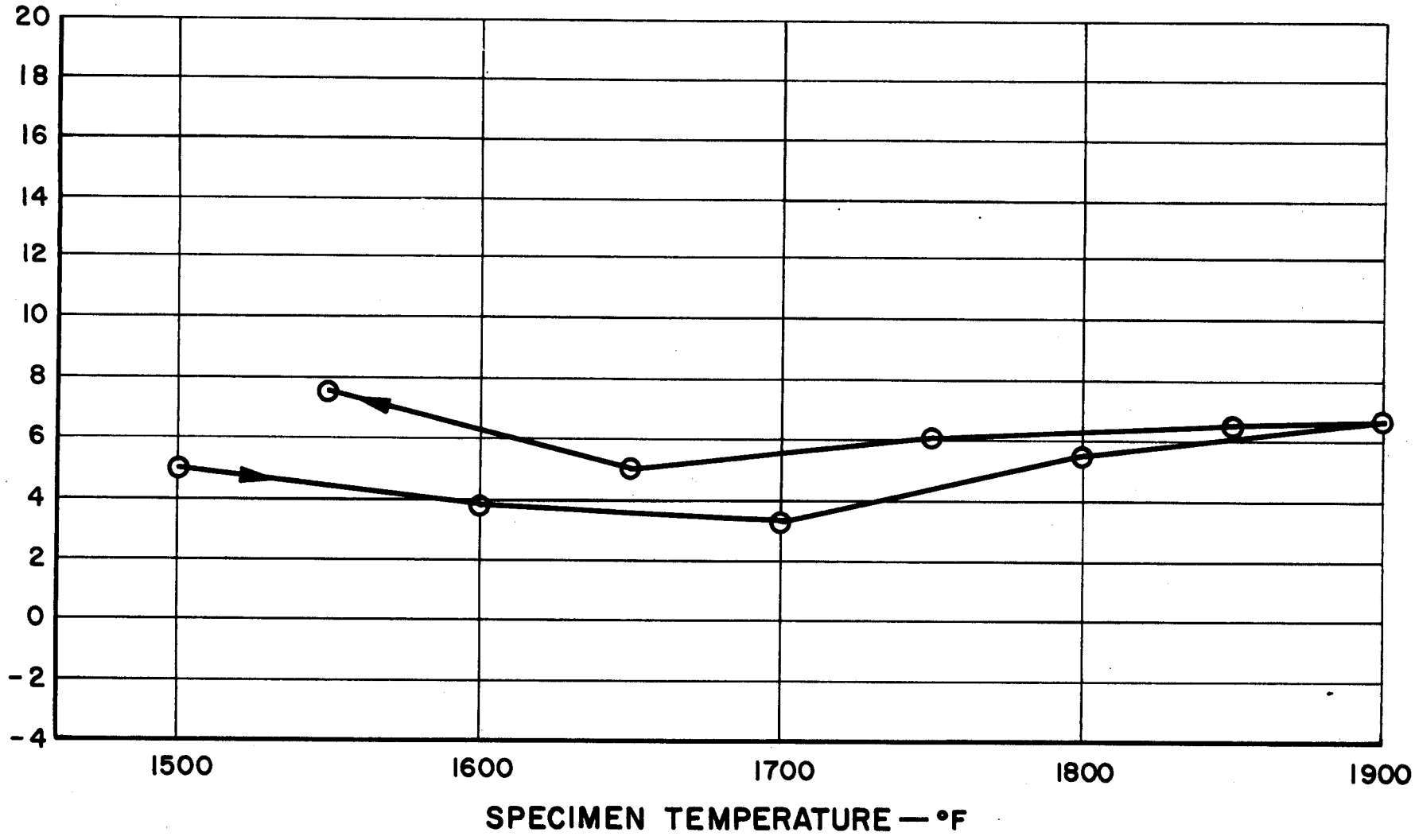

Figure 23
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DIFFERENCE BETWEEN OPTICAL PYROMETER AND THERMOCOUPLE TEMPERATURE INDICATIONS vs. SPECIMEN TEMPERATURE USING TWO CIRCULAR BLACK - BODY HOLES 0.023" IN DIAMETER WITH THE TEST CHAMBER ROTATED 45°

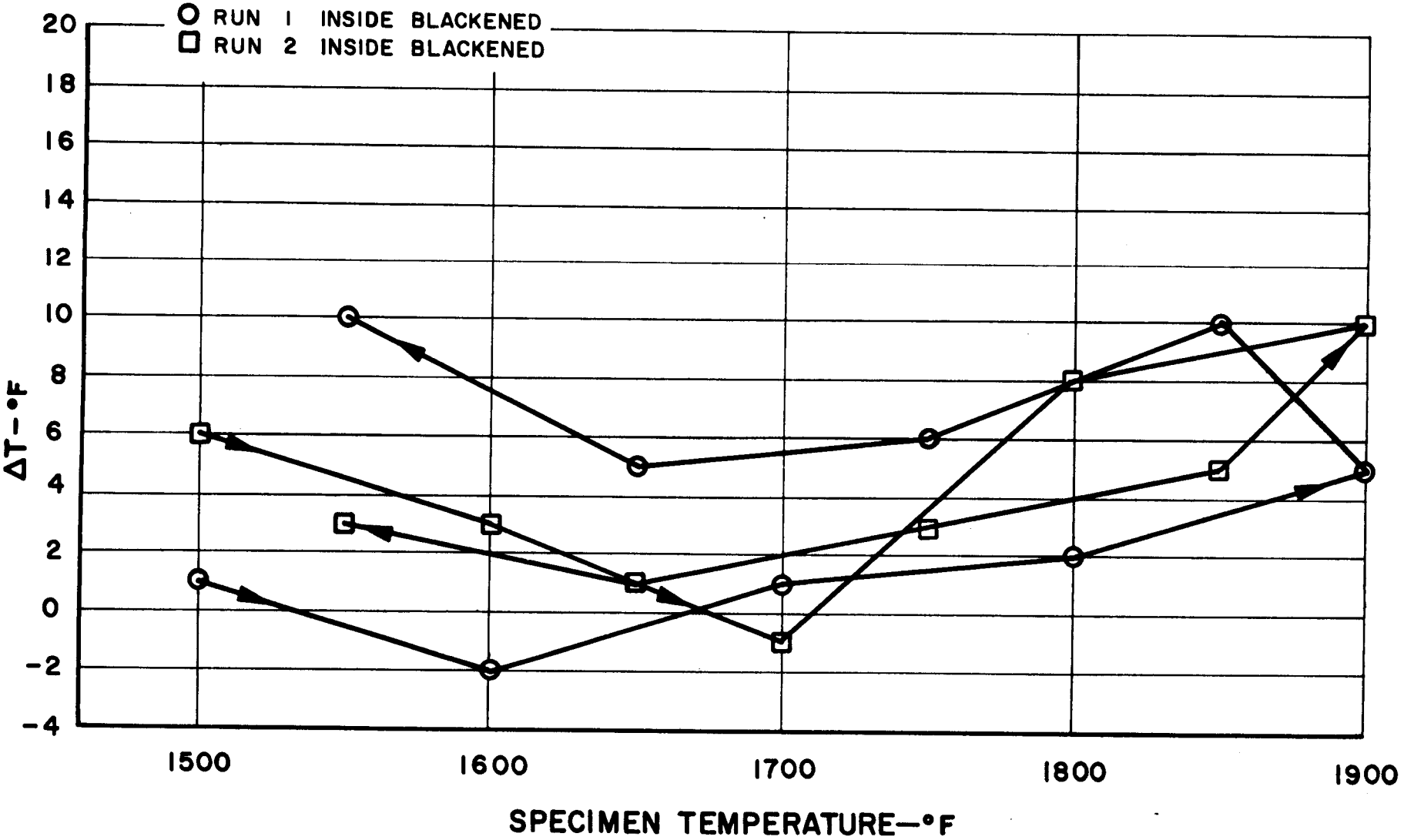

Figure 24
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DIFFERENCES BETWEEN OPTICAL PYROMETER AND THERMOCOUPLE AND BETWEEN THERMODOT AND THERMOCOUPLE TEMPERATURE INDICATIONS USING TWO CIRCULAR BLACK-BODY HOLES 0.023″ IN DIAMETER

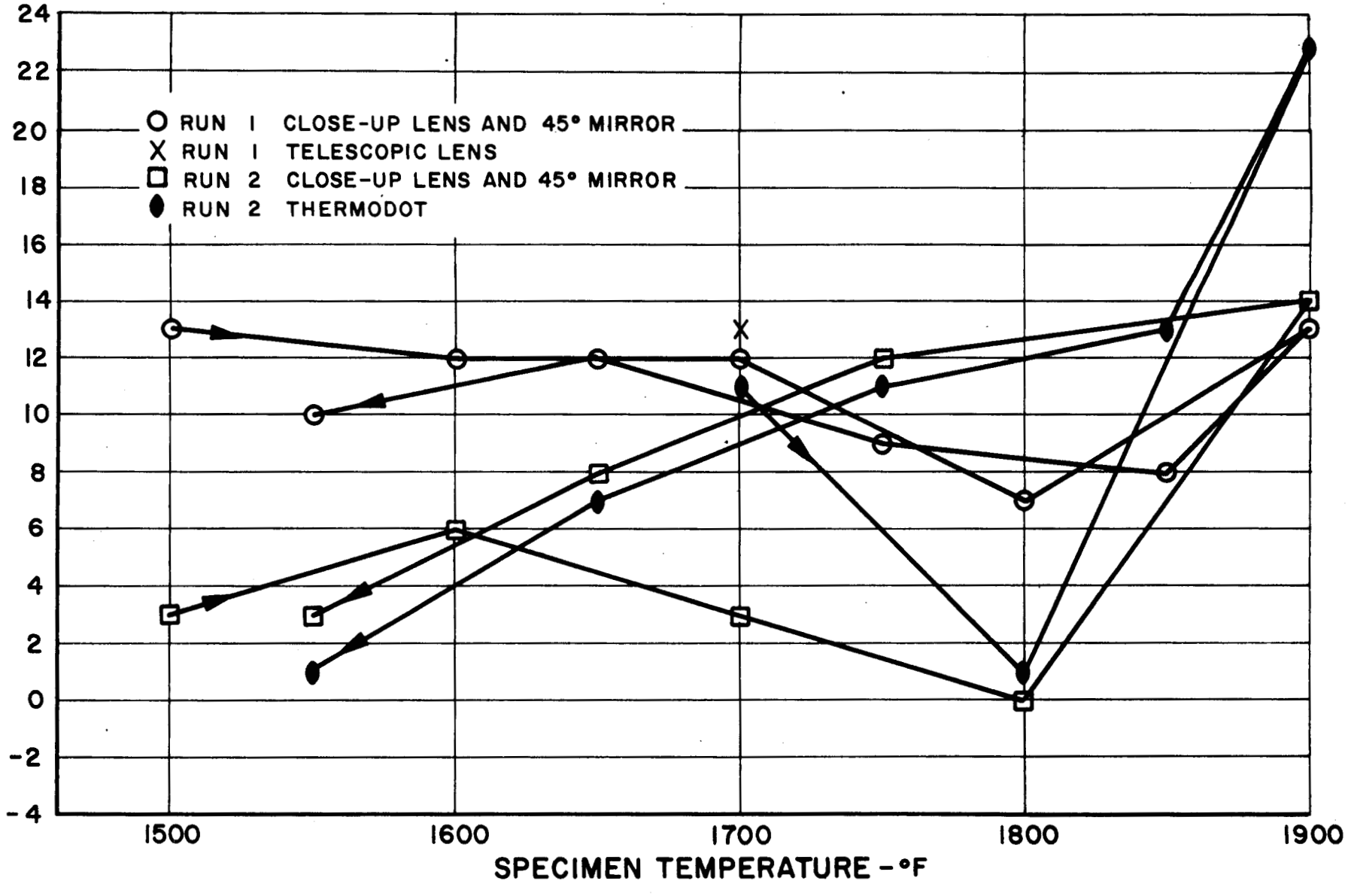

Figure 25
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ERRATA

Please make the following corrections to report PWA-2255 entitled "Progress Report - Determination of the Emissivity of Materials:"

1. Page 20: change optical pyrometer temperature from 500°F to 1500°F

2. Page 27, last sentence: change fin root temperature from 650°F to 700°F

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